

APEX2 User Manual

Version 1.27

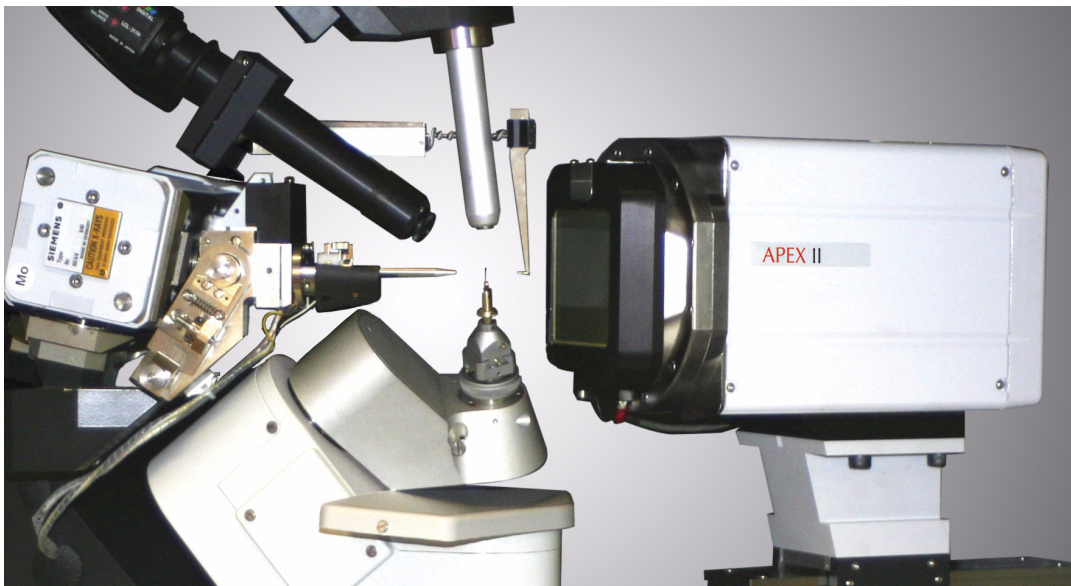
USER MANUAL

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APEX2

USER MANUAL



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1. Introduction

1.1 APEX II Systems for Chemical Crystallography

Bruker AXS Kappa APEX II and SMART APEX II systems are the newest members in the Bruker Nonius product line of instrumentation for single crystal X-ray diffraction. These systems provide the tools for complete small molecule structure determination. The hardware and software are completely redesigned. The software features a new start-to-finish graphical user interface (GUI). The hardware features a new CCD detector based upon four-port readout of a 4K chip and a choice of two goniometers. APEX II systems are enclosed in a radiation safety enclosure system.

The Kappa APEX II system features the Kappa 4-axis goniometer. Two computers are used for experiments. One computer, the server, controls the goniometer. The other computer, the client,

controls all other aspects of the experiment, from data collection through report generation.

The SMART APEX II system is an enhanced version of the SMART APEX fixed-chi system. A single computer controls the data collection, and solution and refinement of the structure.

From a software and operational viewpoint, the APEX II systems use the GUI of the APEX2 software suite to control all operations from crystal screening to report generation for a typical crystallography study. This is a complete departure from the command-driven, functionally separate modules of SMART, SAINTPLUS and SHELXTL. Enhanced versions of the proven and widely accepted programs used by these modules (e.g., SAINT, SADABS, XPREP, XS, XM, XL, etc.) underlie the GUI and provide powerful tools.

From a hardware viewpoint, APEX II systems share common hardware components with other Bruker products. Other members of this new generation of instruments include the D8 ADVANCE and D8 DISCOVER, and the D8 GADDS systems for general diffraction. Documentation on some of these common hardware and software components is available in the user manuals for the D8 family of instruments.

1.2 User Manual Features

This user manual and associated YLID test data are intended to provide you with a step-by-step guide to data collection and processing using the APEX2 software program.

The test data supplied was collected on a Kappa APEX II diffractometer with graphite monochromated molybdenum radiation from a sealed tube generator. The high quality data (resolution=0.75 Å) allows easy refinement of the hydrogen atom positions and determination of the absolute structure of the sample.

NOTE: Before using this manual, check that your system is in proper working order (e.g., the optics and goniometer are aligned) and that the APEX2 suite is properly installed.

1.3 X-ray Safety



WARNING

X-ray equipment produces potentially harmful radiation and can be dangerous to anyone in the immediate vicinity unless safety precautions are completely understood and implemented. All persons designated to operate or perform maintenance on this equipment need to be fully trained on the nature of radiation, X-ray generating equipment and radiation safety. All users of the X-ray equipment are required to accurately monitor X-ray exposure by proper use of X-ray dosimeters.

For safety issues related to the operation and maintenance of your particular X-ray generator, diffractometer and shield enclosure, please refer to the manufacturer's operation manuals or your Radiation Protection Supervisor. The user is responsible for compliance with local safety regulations.

2. Hardware Overview

The two hardware platforms for the APEX II systems are the Kappa APEX II (the four-axis advanced research instrument) and the SMART APEX II (the three-axis laboratory instrument). Software functionality is essentially the same for both platforms.

2.1 System Components

The system (Figure 2.1 and Figure 2.2) consists of the following basic components.

- APEX II CCD detector
- 4-axis Kappa or 3-axis SMART goniometer
- K780 X-ray generator
- Radiation safety enclosure with interlocks and warning lights
- D8 controller
- Refrigerated recirculator for the detector
- Computer(s) (two for the Kappa APEX II and one for the SMART APEX II)
- Video microscope
- Accessories (high- and low-temperature devices)

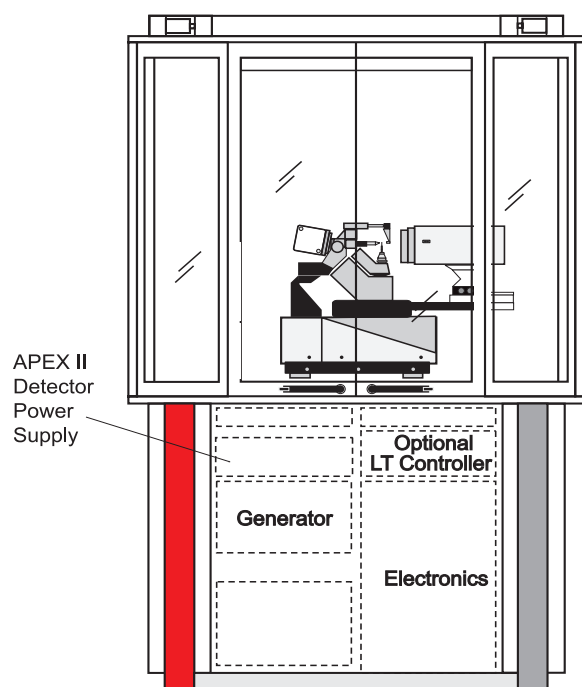


Figure 2.1 - Kappa APEX II system

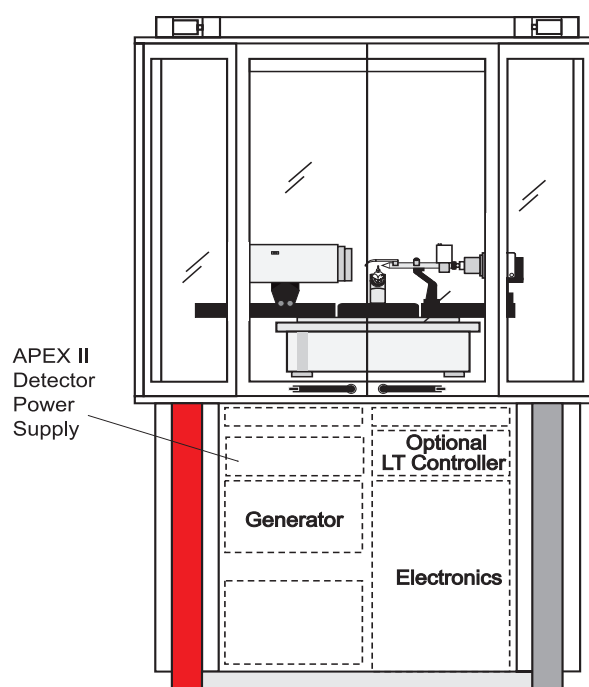


Figure 2.2 - SMART APEX II system

2.1.1 APEX II Detector

The APEX II detector is specific to this system. Status lamps on the top of the detector housing indicate when the detector is on (green) and off (red).

On Kappa APEX II systems, the detector is mounted on a motorized 2-theta track. The camera distance is computer-controlled (a typical distance for the camera is 40 or 50 mm).

On SMART APEX II systems, the detector is mounted on a 2 θ dovetail track. The track has a scale that is calibrated in mm to indicate the distance from the crystal to the phosphor window (a typical distance for the camera is 40 or 50 mm).

An optional motorized DX track is available for the SMART APEX II.

2.1.2 Goniometer

The goniometer module and APEX II detector make up the unique hardware of the system. This is the part of the instrument that actually performs the experiment.

Several components make up the goniometer module with APEX II detector:

- Goniometer (3-axis or 4-axis)
- APEX II detector
- X-ray source (including shielded X-ray tube, X-ray safety shutter, and graphite crystal monochromator)
- K780 X-ray generator
- Timing shutter and incident beam collimator (with beamstop)
- Video camera

Kappa APEX II Goniometer

The Kappa goniometer uses a horizontally oriented Kappa goniometer with 2-theta, omega, kappa and phi drives and a motorized DX track for setting the detector distance. It includes mounting points for the video camera and for optional attachments such as the optional low temperature attachment.

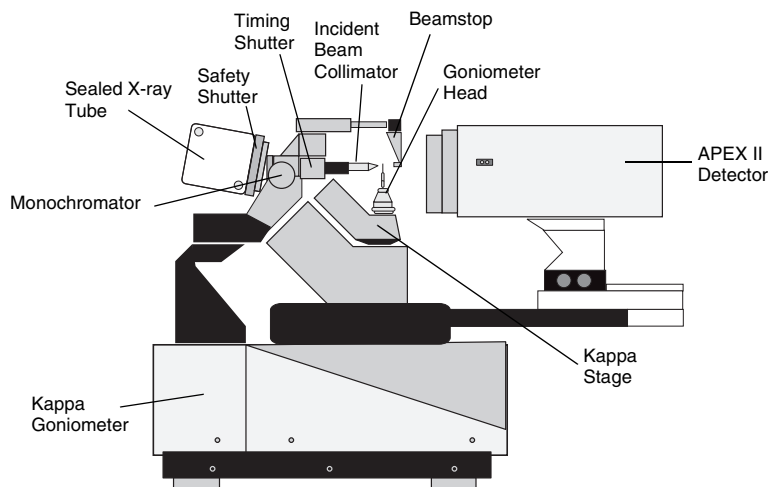


Figure 2.3 - Kappa 4-axis goniometer components

With the kappa angle, the crystal can be oriented at χ from -92° to 92° . This leaves the top of the instrument open for easy access. Kappa can be positioned so that the phi drive, which has unlimited rotation, can be swung under the incident beam collimator, allowing free rotation in omega.

SMART APEX II Goniometer

The SMART APEX II system uses a horizontally oriented D8 goniometer base with 2-theta, omega and phi drives, dovetail tracks for the X-ray source and detector, and an additional mounting track for accessories such as the video camera and optional low-temperature attachment.

The 3-axis system incorporates a fixed-chi stage with chi angle of approximately 54.74° and a phi drive with 360° rotation, which is so compact that it swings under the incident beam collimator, allowing free rotation in omega.

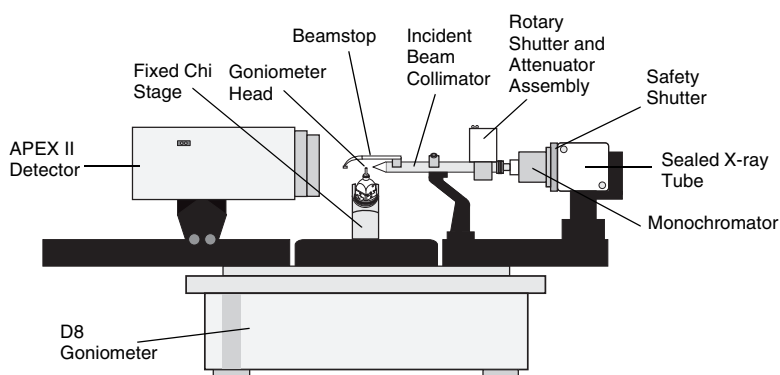


Figure 2.4 - SMART goniometer components

X-ray Source

Three components make up the X-ray source: a shielded X-ray tube, an X-ray safety shutter, and a graphite crystal monochromator.

The sealed tube X-ray source, with a molybdenum (Mo) target, produces the X-ray beam used by the system.

The X-ray safety shutter is built into the X-ray tube shield. The shutter opens upon initiation of a set of exposures and closes upon the end of collection. Status lamps on the shutter housing indicate when the shutter is open (red) or closed (green). The shutter is also interfaced to the controller and to the safety interlocks.

A tunable graphite crystal monochromator selects only the K_{α} line ($\lambda=0.71073 \text{ \AA}$) emitted from the Mo X-ray source and passes it down the collimator system.

K780 X-ray Generator

The K780 X-ray generator is a high-frequency, solid-state X-ray generator that provides a stable source of power for operations up to 60 kilovolts (kV) and 50 milliamperes (mA).

Typical maximum power settings for the APEX II system with a fine focus tube are 50 kV, 40 mA. Either copper or molybdenum tubes may be used on APEX II systems. For both types of tubes, the kV setting should not exceed 50 kV and the power (kV x mA) should not exceed the power rating given on the tube cap.

Because the generator is interfaced to the controller, the power settings can be adjusted within the APEX2 software. This is usually not necessary as the software automatically increases the power to the user-defined values at the beginning of an experiment and lowers them when the instrument is inactive.

Timing Shutter and Collimator

On SMART APEX II systems, the monochromatic X-ray beam then passes through the labyrinth, the timing shutter, and the incident beam collimator before striking the specimen. On Kappa APEX II systems, the monochromatic X-ray beam passes through a small labyrinth, the timing shutter, a secondary labyrinth and the incident beam collimator before striking the sample.

- The labyrinth is a device that ensures that the collimator and shutter are tightly connected to prevent X-ray leakage.
- The timing shutter is a device which precisely controls the exposure time for each frame during data collection. Its status lamps indicate when the shutter is open (ON) and closed (OFF). For SMART APEX II systems, this assembly also houses an automatic attenuator. Kappa APEX II systems do not have an attenuator.
- The incident beam collimator is equipped with pinholes in both the front (near crystal) and rear (near source). These pinholes help

to define the size and shape of the incident X-ray beam that strikes the specimen. (Collimators are available in a variety of sizes, depending on your application.)

- The beamstop catches the remainder of the direct beam after it has passed the specimen. The beamstop has been aligned to minimize scattered X-rays and to prevent the direct beam from hitting the detector. The entire collimator assembly is supported by a collimator support assembly, which has been precisely aligned to guarantee that the X-ray beam passes through the center of the goniometer.

Video Camera

The video camera, an essential part of the system, allows you to visualize the crystal to optically align it in the X-ray beam. It also allows you to measure the crystal's dimensions and index crystal faces. The camera is interfaced to the computer and is operated through the VIDEO program. The VIDEO program includes several computer-generated reticles and scales to make it easy to center and measure the crystal.

2.1.3 Radiation Safety Enclosure with Interlocks and Warning Lights

A common component of all systems in the D8 family is the radiation safety enclosure. This new design is fully leaded (i.e., leaded windows, leaded metal sides and panels) to protect you from stray radiation. The enclosure also includes warning lamps (a government requirement) that alert you when X-rays are being generated. As a special feature, the enclosure also incorporates interlocks for both hardware and software: an automatic system-interruption device that senses when the doors and panels are open and prevents data collection and use of the shutter until you close the doors.

2.1.4 D8 Controller

The D8 controller is an electronic module enclosed in the rack behind the front panel of the instrument. It contains all of the electronics and firmware for controlling the generator, opening the X-ray shutters, and monitoring other instrument functions such as safety interlocks, generator status, and detector status. For SMART APEX II systems, the goniometer is controlled by the D8 controller. For Kappa APEX II systems, there is an additional module, the Kappa controller, for positioning the Kappa goniometer angles and adjusting the detector distance by driving the detector along its track.

2.1.5 Refrigerated Recirculator for the Detector

To minimize dark current in the APEX II detector, dual Peltier devices are used to cool the CCD chip to approximately -58°F (-50°C). The refrigerated recirculator uses an ethylene glycol/water mixture to absorb the heat from the Peltier devices.

2.1.6 Computer(s)

The Kappa APEX II system uses two high-speed computers. The **server** controls the instrument and is used for crystal centering and screening. The **client** collects the data, stores the raw frames, processes the data, and solves and refines the structure. The two computers are linked via a hub and communicate with each other via TCP/IP protocols.

The SMART APEX II system uses a single high-speed computer for control of the experiment, storage of raw frame data, integration of the data, and solution and refinement of the structure.

The computer or computers are often attached to a network of similarly configured computers with access to local and/or network printers.

NOTE: Connection to the external network must be done with care. Consult with local security experts.

2.1.7 Accessories

Various devices can be mounted on the goniometer base. These include optional low- and high-temperature attachments. Both instruments can be used with diamond-anvil cells.

3. Software Overview

This section presents an outline of the system software, including a brief description of the software layout as well as the graphical user interface (GUI).

APEX2 runs on two computers: the server and the client. For SMART APEX II systems, the

server and the client execute on the same computer, but their functionality remains separate. The flowchart in Figure 3.1 shows the software layout. For both Kappa APEX II and SMART APEX II systems, the server and client communicate using TCP/IP protocol.

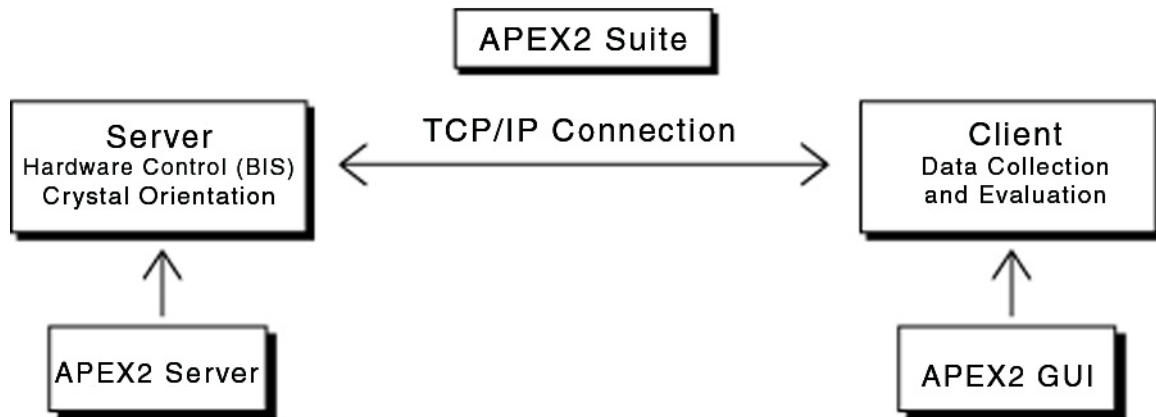


Figure 3.1 - APEX2 software diagram

3.1 The Server Computer

The server computer communicates with the hardware, allowing the user to control the instrument. The server computer runs software for aligning the system, as well as software for aligning and screening samples.

3.1.1 Bruker Instrument Service (BIS)

BIS provides the link between the hardware and software. Once a connection is established, BIS executes hardware commands sent by the APEX2 software. The instrument service can also be used as a service tool, displaying diagnostic messages during operation.

3.1.2 Bruker Control Program (BCP)

BCP is used to configure BIS, as well as to provide instrument control and alignment tools. See the online help within BCP for more information.

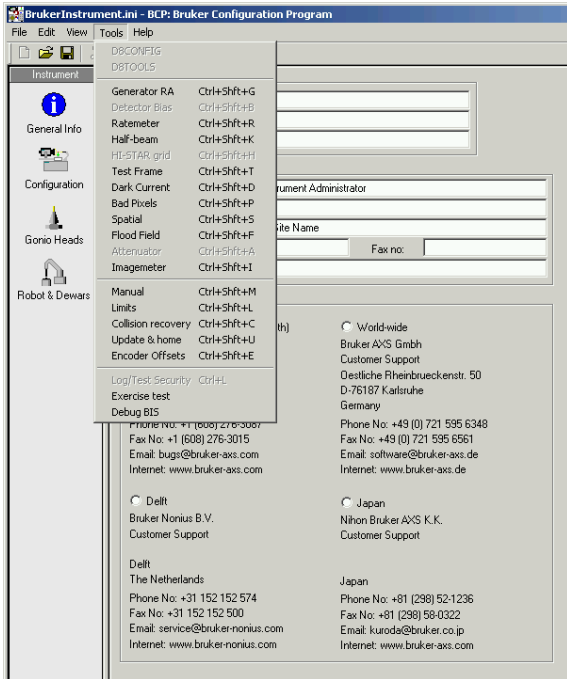


Figure 3.2 - BCP main window

3.1.3 APEX2 Server

The APEX2 Server provides tools for aligning and screening samples. There are two main items: Align Crystal and Simple Scans (see Figure 3.3).

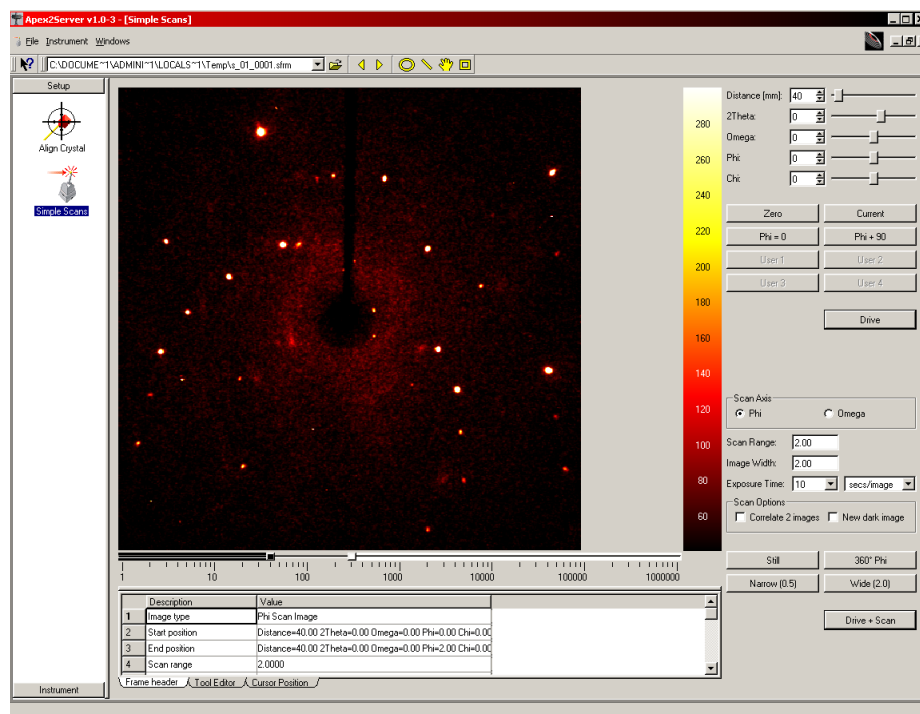


Figure 3.3 - Simple Scans window

3.2 The Client Computer

The client can be any computer on the same network as the server. For SMART APEX II systems, it is usually the same computer as the server. The main portion of the APEX2 suite, the APEX2 client, runs on the client computer. The client is a GUI with multiple plug-ins or modules for different aspects of an experiment. The client includes a database which stores relevant data from each step in the experiment. Details of the functions available in the GUI will be explained in more detail later in the manual.

3.2.1 Database and Database Connection

As currently configured, the database is used internally by the APEX2 Suite and is not available for user customization or manipulation. It must be running before the APEX2 Suite is started and it should be stopped before the computer is shut down (see Section 4.3.2).

The database is used for the storage of data generated by the Bruker APEX2 software.

3.2.2 APEX2 GUI

The APEX2 GUI has one main window (see Figure 3.4). This window is divided into four sections:

- Window Tool Bar
- Tool Icon Bar
- Task Bar
- Task Display Area

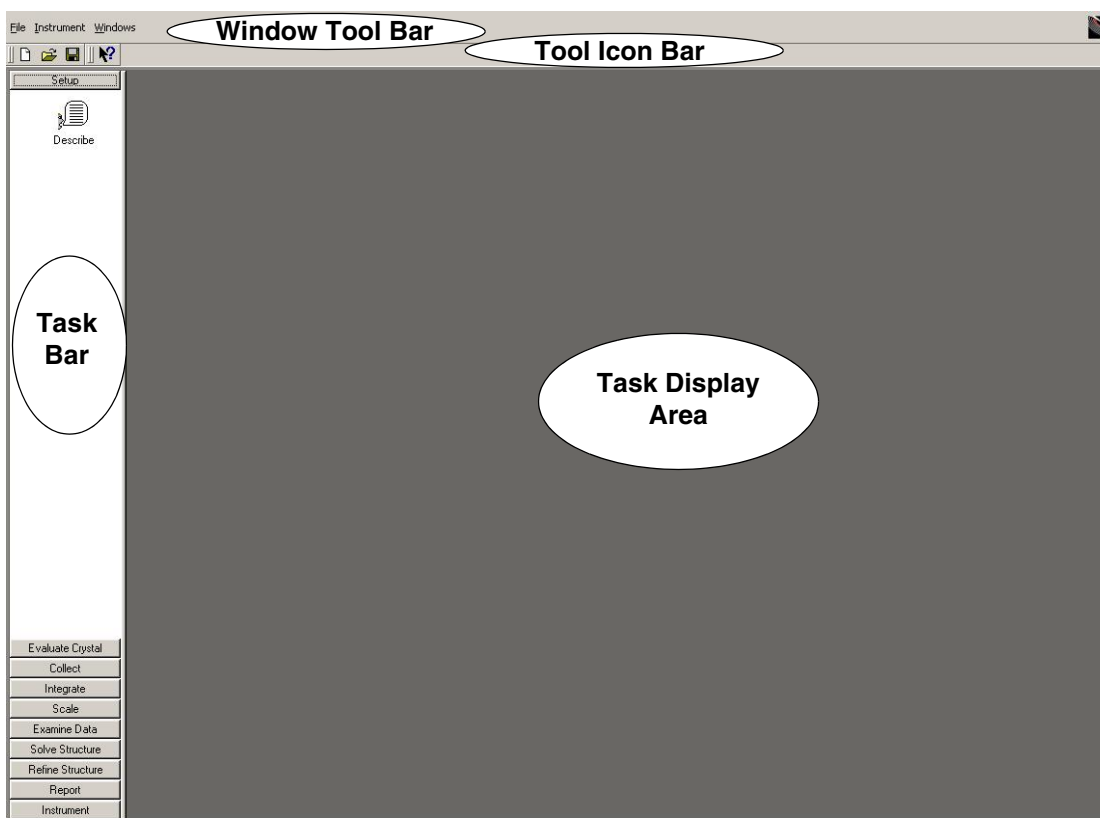


Figure 3.4 - APEX2 GUI

Window Tool Bar

The tool bar provides pull-down menus for a variety of file operations, image tools, and help files.

	Description
[Symbol]	Use this menu to select the following: Restore, Move, Size, Minimize, Maximize, and Close.
File	Use this menu to select the following: Login, Logout, New, Open, Save, Close, Import (Spatial), Export (.p4p file) and Exit.
Instrument	Use this menu to select the following: Connection, Status, Toggle Shutter and Abort.
Windows	Use this menu to select the following: Cascade and Tile.
RLATT (available when you select Reciprocal Lattice Viewer)	Use this menu to select the following: Rotate, Edit, Orientation, Unit Cell Tool, Measure Distance, Measure Angle and Visualization.
View (available when you select Data Collection Strategy)	Use this menu to select the following: Detailed Strategy.

Table 3.1 – Window tool bar options

Tool Icon Bar

The tool icon bar provides shortcuts to the options available through the window tool bar.

Icon	Description
	Create a new file.
	Open a file.
	Save a file.
	“What’s this?” Context-sensitive help.
	Open an image.
	Select the first image in a run. This icon is visible only when an image is displayed.
	Select previous image. This icon is visible only when an image is displayed.

Table 3.2 – Tool icon bar options

Icon	Description
	Select next image. This icon is visible only when an image is displayed.
	Select the last image in a run. This icon is visible only when an image is displayed.
	Go down one run.
	Go up one run.
	Draw a resolution circle. This icon is visible only when an image is displayed.
	Draw a plotting line. This icon is visible only when an image is displayed.
	Change the part of the image displayed while zoomed in. This icon is visible only when an image is displayed.

Table 3.2 – Tool icon bar options

Icon	Description
	Select a region of the image. This icon is visible only when an image is displayed.

Table 3.2 – Tool icon bar options

Task Bar

The task bar provides menus for all of the options in the APEX2 Suite: crystal evaluation and indexing (Evaluate Crystal), data collection (Collect), data processing (Integrate and Scale), and instrument setup (Instrument).

Setup



Describe - Specify crystal size, color, shape, etc.



Center - Perform crystal centering functions.

Evaluate Crystal



Determine Unit Cell - Determine unit cell and Bravais lattice type.



Reciprocal Lattice Viewer - 3D visualization of lattice projected in reciprocal space.



View Images - View collected frames.

Table 3.3 – Task bar options

Collect



Data Collection Strategy - Simulated data collection and determination strategy.



Experiment - Sequence editor for data collection experiments.



Oriented Scans - Measure different images with the crystal aligned along the axes.

Integrate



Integrate Images - Integration of different data.

Scale



Scale - Scale intensities and perform absorption correction.

Table 3.3 – Task bar options

Examine Data



XPRep (Space Group Determination) - Run XPRep.



Precession Images - Create synthesized precession images based on measured frames.

Solve Structure



Structure Solution - Solve the phase problem to get an initial model.

Refine Structure



Structure Refinement - Use least squares to improve the model.

Report



Run XCiF to generate a report.

Table 3.3 – Task bar options

Task Display Area

The Task Display area is the main area for tasks, user input, and selected output. This area displays images, the reflections used in indexing, and the observed and predicted diffraction patterns. It also displays the runs for data collection and solution and refinement. (For version 1.22,

space group determination, SaintChart output, XSHLL refinement, and XCIF report generation do not use the Task Display Area; they open in a new window). All other plug-ins open in the Task Display area of the GUI.

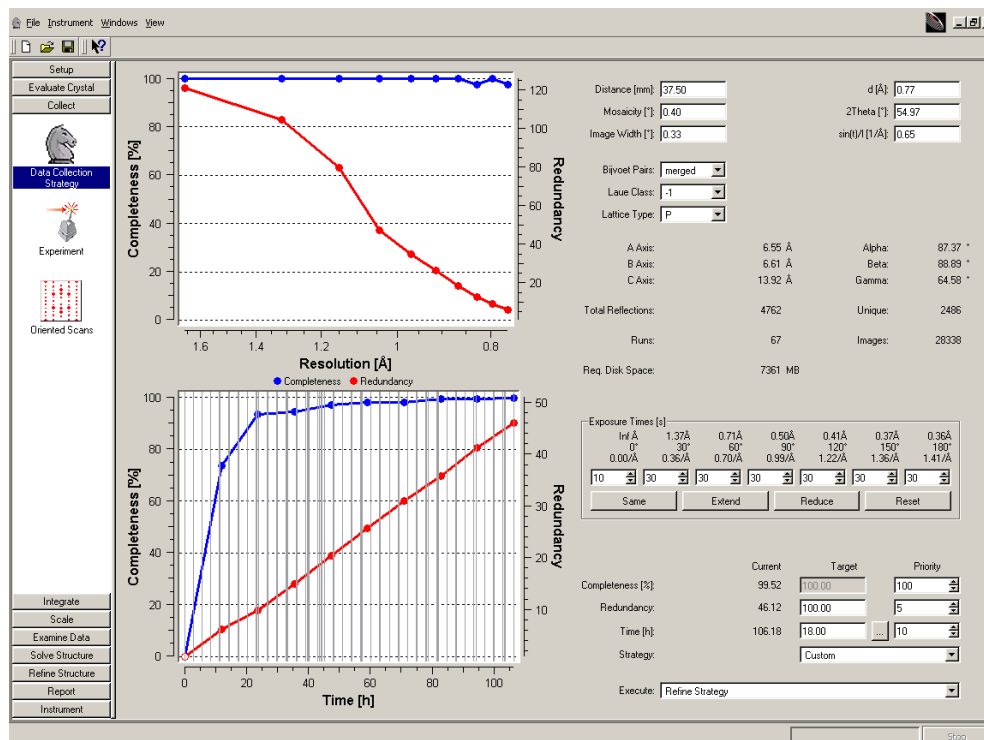


Figure 3.5 - The Task Display area showing COSMO

4. Program Start-Up and Shutdown

As mentioned previously, the APEX2 Suite is composed of several programs. All of the programs are started in a similar fashion. For ease of use there is usually a desktop icon for the folder containing these programs, and desktop icons linked directly to these programs. However, the Start > Programs > Bruker ... path is always available. This more explicit method will be used in this discussion.

4.1 Server Computer Start-Up

Two programs must be running: Bruker Instrument Service and APEX2 Server.

NOTE: For Kappa APEX II systems, the programs will be on the server computer in the goniometer cabinet. For SMART APEX II systems, there is typically only one computer.

4.1.1 Starting Bruker Instrument Service (BIS)

1. Click on Start > Programs > Bruker AXS Programs > Bruker Instrument Service or click on the BIS icon on the desktop.

After a brief initialization period, a window will appear (see Figure 4.1). On Kappa APEX II systems, the goniometer will move to reference positions.

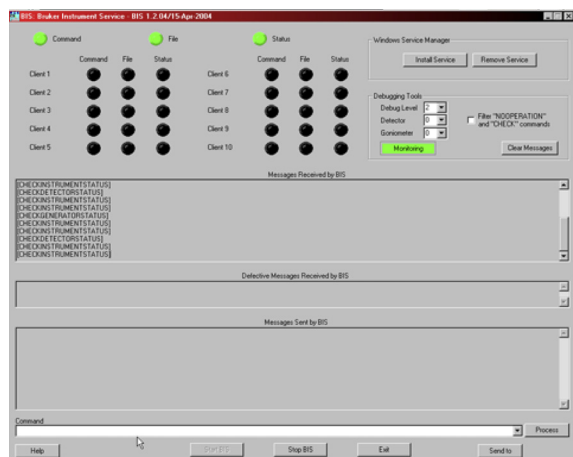


Figure 4.1 - The BIS window

If a small pop-up window appears that says “This second instance of BIS is exiting” (see Figure 4.2), BIS was already running. Click on OK to clear this informational message.



Figure 4.2 - BIS exiting message

NOTE: With a Kappa APEX II, the kappa goniometer will home and the kappa server will activate when BIS is started. This may take a minute or two. The Kappa server is a service tool and should not be used to control the instrument.

4.1.2 Starting the APEX2 Server

1. Click on Start > Programs > Bruker Nonius Programs > APEX2 Server or click on the APEX2 Server icon on the desktop.

A window will appear (see Figure 4.3).

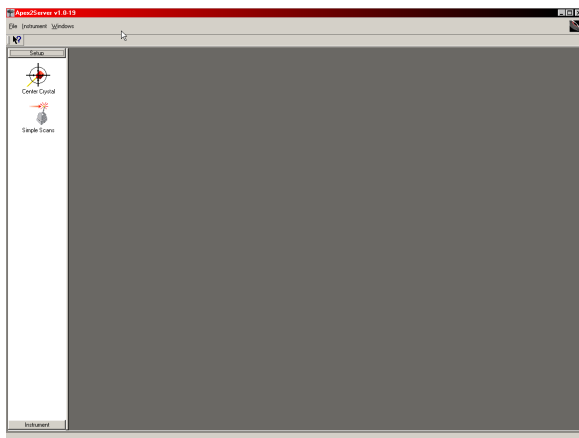


Figure 4.3 - Initial APEX2 Server window

2. In the top left corner, click on the Instrument pull-down menu (see Figure 4.4).

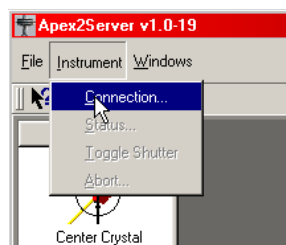


Figure 4.4 - Connecting to the instrument

3. Click on Connection and a new window will appear (see Figure 4.5). The name of your server should already be filled in.
4. Click on Connect.

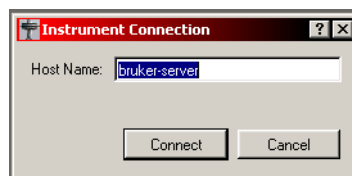


Figure 4.5 - Connection window

NOTE: If the host name is wrong, then the instrument is not properly configured and you should consult your system manager. (It is possible to configure the instrument to automatically connect so that this window will not appear). This is discussed in Appendix E: Config.

4.2 Client Computer Start-Up

On the client computer, two programs are also required: the database and APEX2. It is best to start the database before starting APEX2.

NOTE: For the SMART APEX II, there is typically only one computer for the client and server software.

4.2.1 Starting the Database

- 1a. For Windows systems, click on Start > Programs > Bruker AXS Programs > Start Database or click on the Start Database icon on the desktop.



- 1b. For Linux systems, open a terminal window and type

```
bnrun startdb
```

or click on the Start Database icon.

A window should appear that says the database system is ready.

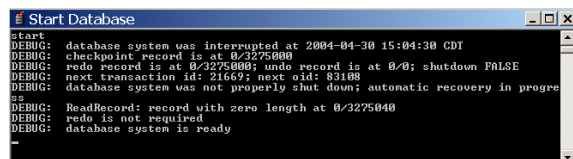


Figure 4.6 - The database is ready

You can minimize this window. If the database has not previously been closed properly (e.g., after a power failure), a window will appear (see Figure 4.7) that states that another postmaster is running. If this happens, stop the database and then start it again.



Figure 4.7 - Database failure message

4.2.2 Starting APEX2

- 1a. For Windows systems, on the client computer click on Start > Programs > Bruker Nonius Programs > APEX2 or click on the APEX2 icon on the desktop.



- 1b. For Linux systems, open a terminal window and type

```
bnrun apex2
```

or click on the APEX2 icon.

2. A window will prompt you to log in to the database by entering a user name and password (see Figure 4.8).
- 2.1 If the system manager has set up the system to automatically enter the user name and password, step 2 is skipped.

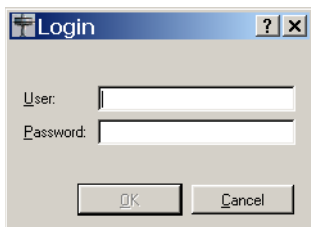


Figure 4.8 - Login request

3. An empty start-up window will appear (see Figure 4.9).

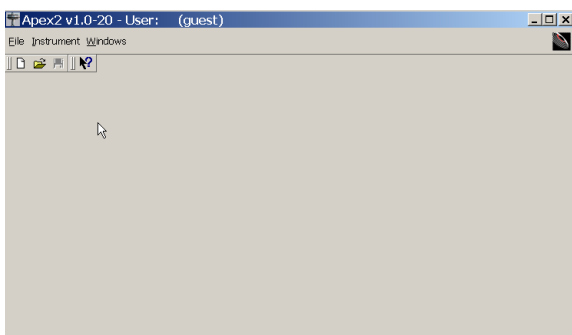


Figure 4.9 - APEX2 start-up window

4. Click on File.

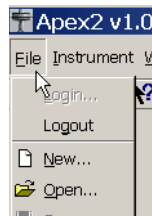


Figure 4.10 - File menu

5. Use the options in this menu to create a new project or to open an existing project.
6. If the window in Figure 4.11 appears, then APEX2 thinks the database is already in use. Answer "Yes" to close the window.

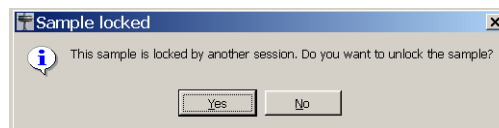


Figure 4.11 - Sample locked window

4.3 Client Computer Shutdown

NOTE: The order of stopping these programs is important. If you attempt to close the database before APEX2 is stopped, the database will remain open until APEX2 is stopped.

4.3.1 Stopping APEX2

1. For Windows or Linux systems, click on the X in the upper right corner of the window or click on File > Exit in the upper left. It is not necessary to disconnect from the instrument.

4.3.2 Stopping the Database

- 1a. For Windows systems, click on Start > Programs > Bruker Nonius Programs > Stop Database or click on the Stop Database icon.



- 1b. For Linux systems, in a terminal window enter

```
bnrun stopdb
```

or click on the Stop Database icon.

A window will appear and quickly disappear, and the Start Database window will close.

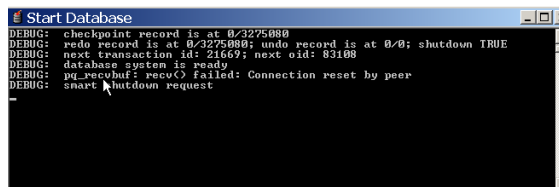


Figure 4.12 - Stop database screen

NOTE: Occasionally the windows won't disappear and the Start Database window will display a "smart shutdown request" (see Figure 4.12). This message means that the database is waiting to close until applications that it might write to are closed. Exit APEX2 to solve this problem. If the message still appears, use the Task Manager to check for other processes that may still be running (e.g., COSMO).

4.4 Server Computer Shutdown

Stop APEX2 Server before BIS. It is acceptable for the order to be reversed. Generally, BIS is never stopped.

4.4.1 Stopping the APEX2 Server

1. Click on the X in the upper right corner of the window or click on File > Exit in the upper left. It is not necessary to disconnect from the instrument.

4.4.2 Stopping BIS

It is almost never necessary to stop and exit BIS. If necessary, click on the Stop BIS button on the bottom of the BIS window and then click on the Exit button at the bottom of the window.

5. Crystal Orientation

We are now ready to begin data collection with the instrument. It is assumed that your system manager has set up the system properly and that all system default parameters have been set appropriately.

The data collection process is divided into five steps, which will be covered in Section 5 and Section 6. The steps in Section 5 are performed using the APEX2 Server software on the server computer. The steps in Section 6 are performed using the APEX2 program on the client computer.

See Section 5 for:

1. Centering/aligning the crystal on the diffractometer (from the APEX2 Server—the Center module)

2. Crystal quality check (from the APEX2 Server—the Simple Scans module)

See Section 6 for:

3. Cell determination (from APEX2—the Cell Determination module)
4. Data collection setup (from APEX2—the Strategy module)
5. Data collection (from APEX2—the Experiment module)

The first steps—mounting, aligning and screening a crystal—are performed on the server computer.

5.1 Mount the Goniometer Head on the Instrument

1. Open the enclosure doors. Push either of the rectangular green Open Door buttons on the side posts. This will release the door locks for approximately five seconds. During this time, pull out on one or both of the handles to physically open the doors.
2. In the APEX2 Server GUI, under Setup click on Center Crystal.

The centering buttons will appear and the video window will open.

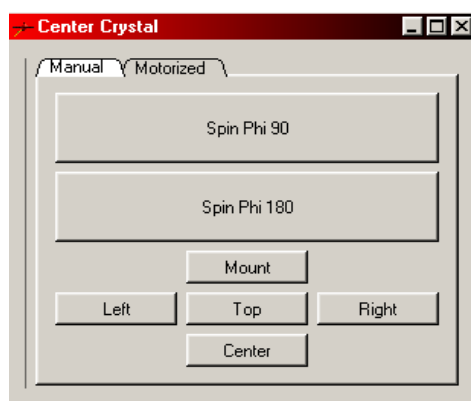


Figure 5.1 - The Center buttons

The bottom five buttons will drive the goniometer to various pre-defined positions that are designed to simplify crystal centering. The top two buttons will drive phi by either 90 or 180 degrees.

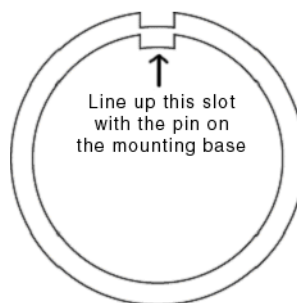
3. Click on Mount to mount the goniometer head.
4. Carefully remove the goniometer head containing the crystal from its case.



CAUTION

Use extreme care when handling the goniometer head to prevent damage to the sample on the end of the small glass fiber.

5. Place the goniometer head onto its base on the phi drive. Line up the slot on the bottom of the goniometer head with the pin on the mounting base (see Figure 5.2).



View of the bottom of the goniometer head

Figure 5.2 - View of the bottom of the goniometer head

6. Screw the head's collar to the base so that the head does not move. Do not overtighten it (finger-tighten only).

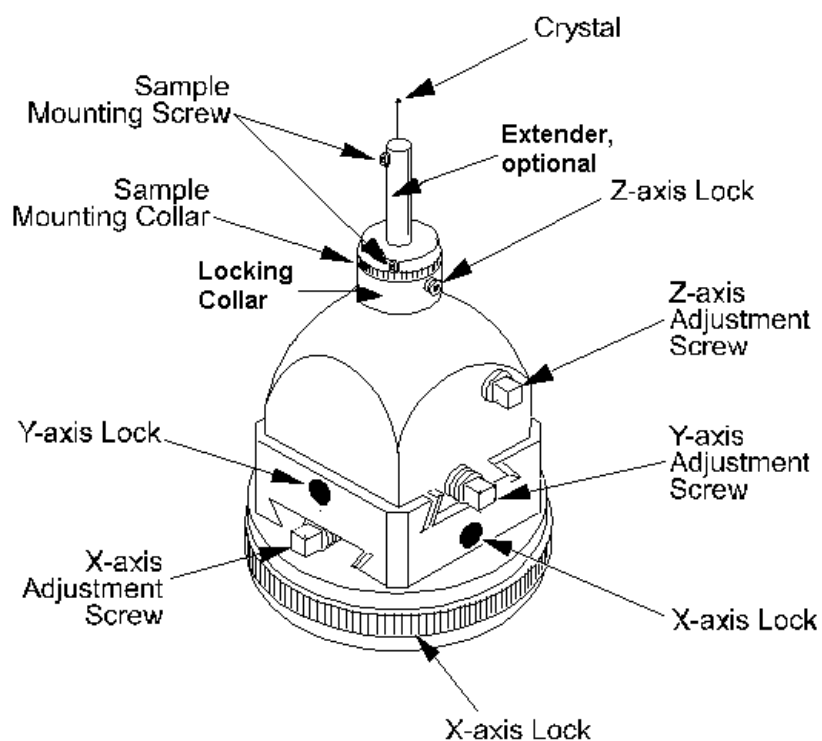


Figure 5.3 - Huber goniometer head in detail

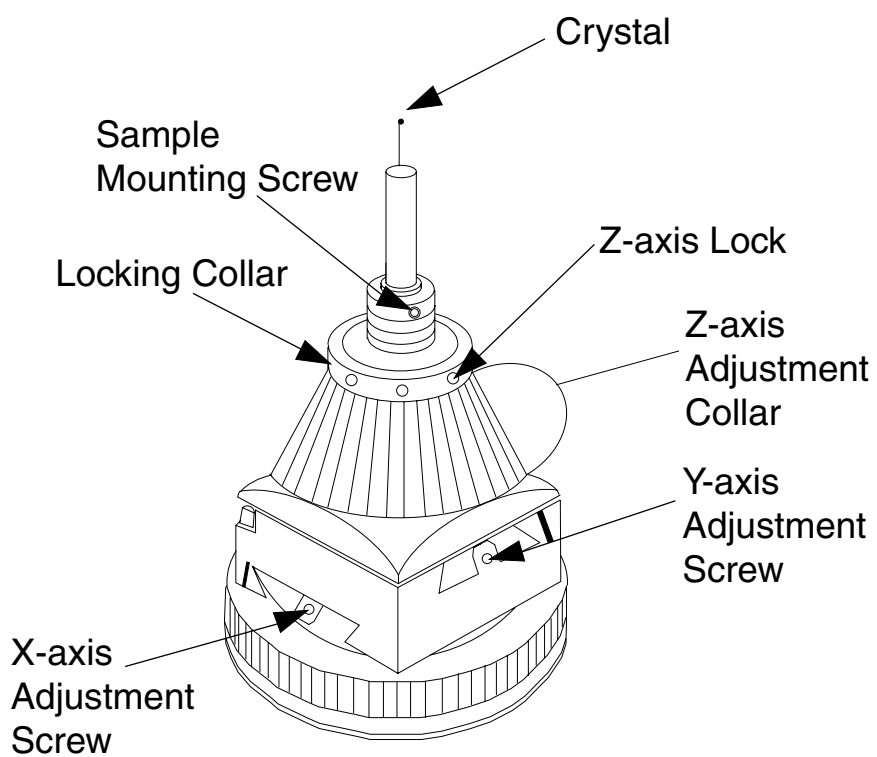


Figure 5.4 - Standard goniometer head in detail

5.2 Center and Align the Sample

To obtain accurate unit cell dimensions and to collect good quality data, align the center of the sample with the center of the X-ray beam and maintain this alignment for the entire experiment. Your video camera should be aligned so that the crosshairs of the video camera coincide with the center of the goniometer and the center of the X-ray beam (see manual M86-Exx024 for instructions on aligning the microscope to the center of the instrument). If the microscope is not centered, you can still align the sample—the key to crystal centering is that the crystal stays in the same place in the microscope's field of view in all orientations.

NOTE: Use the thin end on the goniometer wrench to unlock the X, Y and Z locks at the beginning of the centering process and to lock them at the end—locking needs only a very slight touch. The other end of the wrench is used to move the adjustment slides. Do not overtighten.

NOTE: Centering is often easier if the crystal is rotated to give a good view before the actual centering process is started (e.g., down an edge for a plate). To do this, drive to one of the centering positions, loosen the screw that locks the crystal mounting pin, rotate the crystal to a suitable orientation and then tighten the screw again.

5.2.1 For a Kappa APEX II System

1. Click the Center button—the crystal and goniometer head will be positioned perpendicular to the microscope. To center the sample, make adjustments in the height with the Z-axis adjustment and with the translation screw that faces the front of the diffractometer.

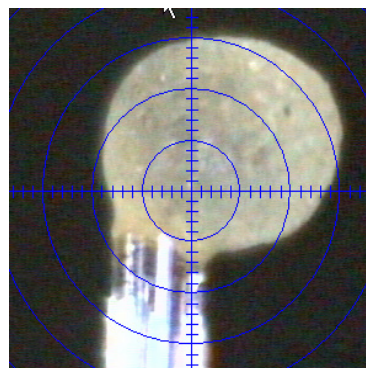


Figure 5.5 - Crystal initially mounted

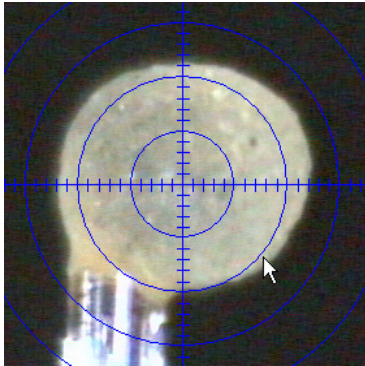


Figure 5.6 - Crystal centered

2. Adjust the height with the Z-axis screw.
3. Adjust the translation with the X- or Y-axis screw, whichever is facing you.
4. Click Spin Phi 90 and adjust the crystal position using the X- or Y-axis screw. (Adjust to remove half of the difference.)

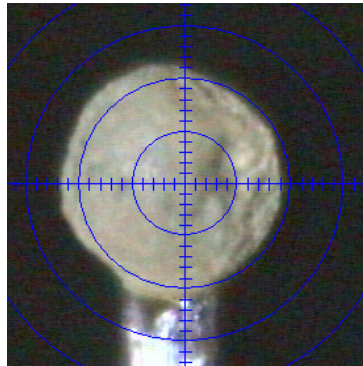


Figure 5.7 - Spin Phi 90

5. Click Spin Phi 180 and adjust the screw facing you, as needed. (Adjust to remove half of the difference.)

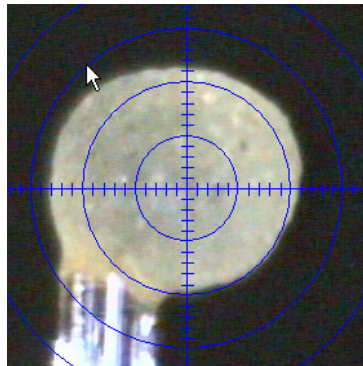


Figure 5.8 - Spin Phi 180

6. Click Spin Phi 180 and Spin Phi 90, making adjustments until the crystal stays in the same place in the microscope.
7. As needed, repeat step 2 through step 5 to keep the crystal in the same place in the microscope.
8. Click the Left button and note the height. The goniometer drives to place the fiber horizontal and to the left.
9. Click the Right button and check that the crystal height stays in the same place in the microscope.
 - 9.a If the height is in the same place, you are done.
 - 9.b If the height is not in the same place, adjust to remove half of the difference and repeat step 8 and step 9.

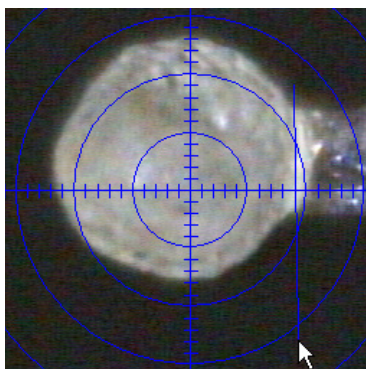


Figure 5.9 - Check the crystal height

10. Click the Top button. Click Spin Phi 180 a few times to verify that the sample stays in the same position. If it is not centered, go back to step 2.

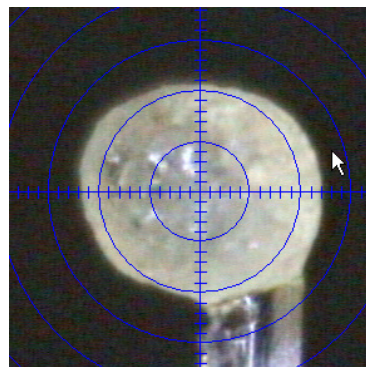


Figure 5.10 - The crystal is centered

11. Go back to the Center position.

The crystal is now centered on the instrument. All of the next steps are performed with APEX2 on the client computer.

5.2.2 For a SMART APEX II System

NOTE: If the image of the crystal is difficult to see, illuminate the sample with a high-intensity lamp and/or temporarily place a light-colored piece of paper on the front of the detector.

1. Click the Right button. The crystal and goniometer head will be positioned perpendicular to the microscope. To center the sample, make adjustments to the height with the Z-axis adjustment.

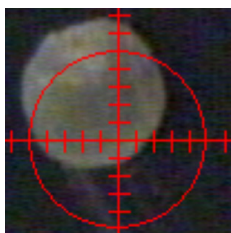


Figure 5.11 - Initial mounted crystal

2. Click the Center button. Move the crystal so that it is centered in the microscope reticle by adjusting the X- or Y- axis translation adjustment screw that is perpendicular to the microscope axis and facing you (see Figure 5.3 and Figure 5.4).

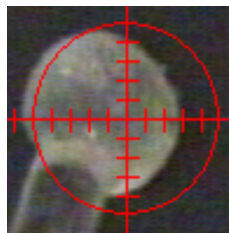


Figure 5.12 - Initial center position



Figure 5.13 - Initial X- or Y-axis (translation) adjustment

3. Click Spin Phi 90. Remove half of the difference with the adjustment screw that is facing you.



Figure 5.14 - Spin Phi 90

4. Click Spin Phi 180. Remove half of the difference with the adjustment screw that is facing you.
5. Click Spin Phi 180 again.
 - 5.1 If the crystal is centered, click Spin Phi 90.
 - 5.2 If the crystal is not centered, adjust to remove half of the difference and click Spin Phi 180. Repeat until the crystal is centered. Click Spin Phi 90.
 - 5.3 If centered, adjust the height. If not centered, repeat steps 2 through 5 until it is centered.



Figure 5.15 - Height adjusted

6. Click the Left button. Adjust to remove half of the difference. Adjust the height.



Figure 5.16 - Check Left

7. Click the Right button. Adjust the height. Adjust to remove half of the difference.

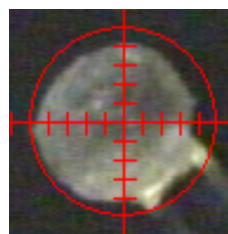


Figure 5.17 - Check Right

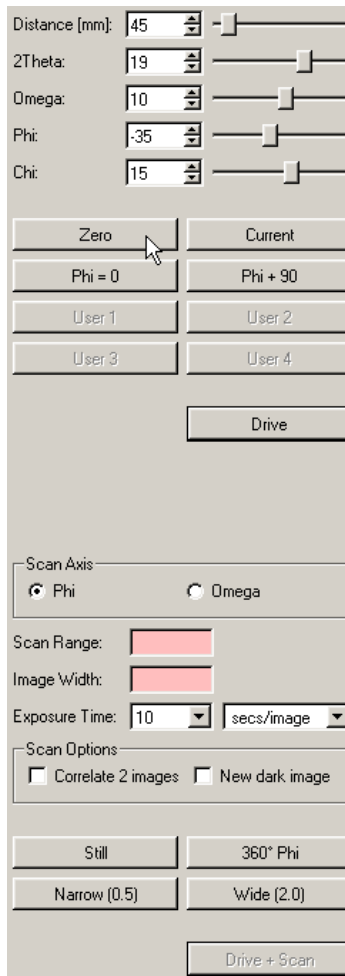
8. If a height adjustment was made in step 6 or 7, repeat those steps to check the height. If the height is adjusted, repeat steps 2 to 5 to check the centering. If no height adjustment was made, the crystal is centered.

The crystal is now centered on the instrument. All of the next steps are performed with APEX2 on the client computer.

5.3 Simple Scans

The Simple Scan plug-in provides the tools for rapid screening of the sample to check sample quality. It allows the user to quickly set up scans to measure a 360-degree phi rotation as well as still, thin (0.5 degree) and thick (2.0 degree) images.

1. Click on the Simple Scan icon. The menu shown in Figure 5.18 will open.



The Simple Scans menu interface is a vertical panel with various controls. At the top, it features five rows of controls for Distance (mm), 2Theta, Omega, Phi, and Chi, each with a numeric input field and a slider. Below these are two columns of buttons: 'Zero' and 'Current' in the first row, 'Phi = 0' and 'Phi + 90' in the second, 'User 1' and 'User 2' in the third, 'User 3' and 'User 4' in the fourth, and a single 'Drive' button centered in the fifth row. Further down is a 'Scan Axis' section with radio buttons for 'Phi' (selected) and 'Omega'. Below that are 'Scan Range' and 'Image Width' sections, each with a red rectangular input field. The 'Exposure Time' section has a dropdown menu set to '10' and a unit dropdown set to 'secs/image'. The 'Scan Options' section contains two checkboxes: 'Correlate 2 images' and 'New dark image'. At the bottom, there are four buttons arranged in a 2x2 grid: 'Still' and '360° Phi' in the top row, 'Narrow (0.5)' and 'Wide (2.0)' in the bottom row, and a large 'Drive + Scan' button centered at the very bottom.

Figure 5.18 - Simple Scans menu

The sliders and data boxes at the top can be used to position the detector.

The buttons in the middle provide easy access to common movements.

There are four possible user-defined buttons.

The Drive button initiates the requested movement. If it is gray, an impossible movement has been requested.

The buttons and boxes at the bottom set up scans. In Figure 5.18, the Drive + Scan button is grey and therefore inactive because no scan has been requested.

2. Click on Zero and then on Drive.
3. Set the distance.
 - 3.1 On Kappa APEX II systems, check that the moveable beamstop is pushed in and set the desired position (typically 45 mm) for Distance in the data window.
 - 3.2 On SMART APEX II systems with movable DX, set the desired position (typically 50 mm) for Distance in the data window.
 - 3.3 On SMART APEX II systems with fixed DX, check that the distance displayed is the same as the actual distance in mm on the detector arm.

4. Click 360degree Phi and set the desired exposure time. The default of 15 seconds is usually sufficient.
5. Click "Drive + Scan". Since these are evaluation scans, there is no need to request correlated images or new darks. The resulting Phi 360° image is shown in Figure 5.19. The crystal diffracts nicely with lots of sharp spots. Figure 5.23 shows a Phi 360° scan with a bad crystal.

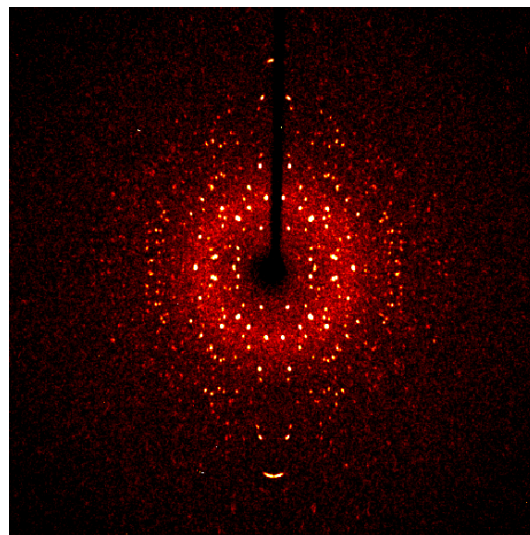


Figure 5.19 - A 360° Phi scan on a good quality crystal

6. Click on Wide (2.0), change the scan range to 2.0 and set the desired exposure time. A time of 5 to 15 seconds is usually sufficient.

7. Click “Drive + Scan”. The resulting 2-degree scan is shown in Figure 5.20. The spots are sharp and clean. There are no peaks that are very close together. Figure 5.24 shows a 2-degree scan with a bad crystal.

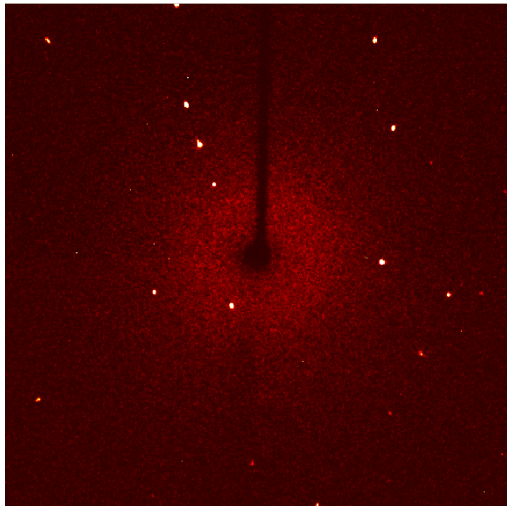


Figure 5.20 - A 2° phi scan on a high quality crystal. The spots' shapes are well-defined and the spots are well-separated.

8. Click “Phi + 90” in the middle row of boxes.
9. Click “Drive + Scan”. The resulting 2-degree scan is shown in Figure 5.21. This image is measured 90 degrees from the previous one giving a view of the diffraction pattern from a different (perpendicular) direction. Figure 5.25 gives a similar view for the poor crystal.

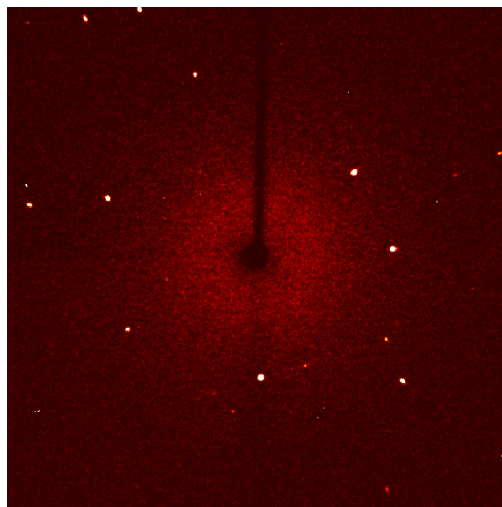
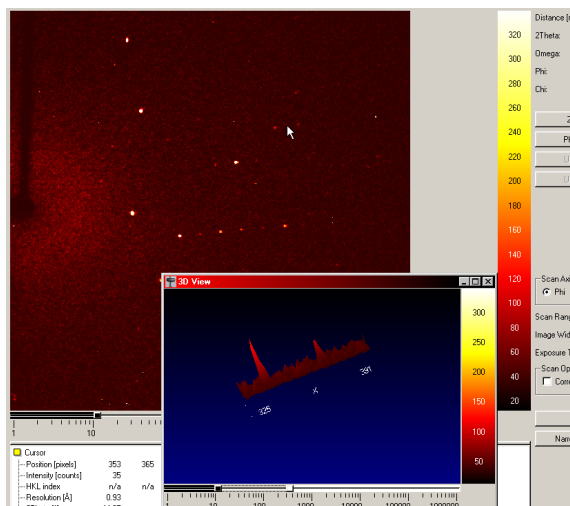


Figure 5.21 - A 2° phi scan at plus 90 in phi on a high quality crystal. The spots' shapes are well-defined and the spots are well-separated.

10. Set 2Theta to -30. This will allow evaluation of the diffraction at higher angles.

11. Click “Drive + Scan.” The resulting image is shown in Figure 5.22.



5.4 Examples of Poor Screening

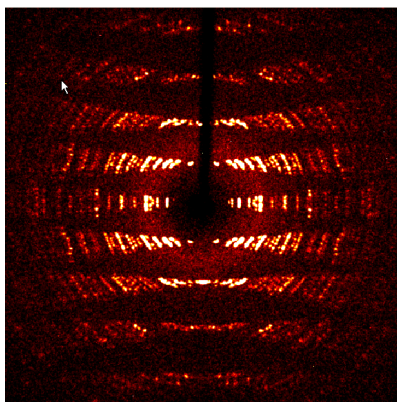


Figure 5.23 - A 360° phi scan on a poor quality crystal. The spot shape is poor and the spots tend to run together. The obvious bands on the image suggest that the crystal is nearly aligned on an axis.

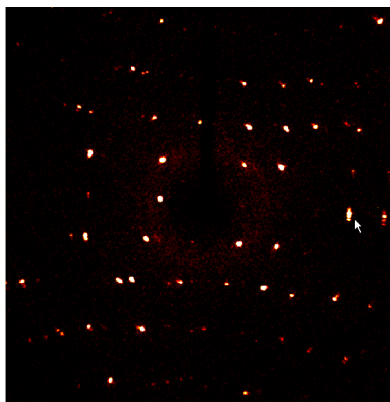


Figure 5.25 - A 2° phi scan on a poor quality crystal at plus 90 in phi. The spot shape is poor and the spots are very close together.

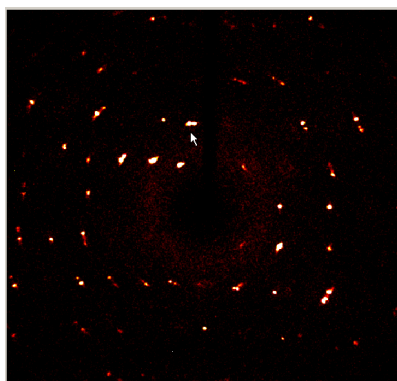


Figure 5.24 - A 2° phi scan on a poor quality crystal. The spot shape is poor and the spots are very close together.

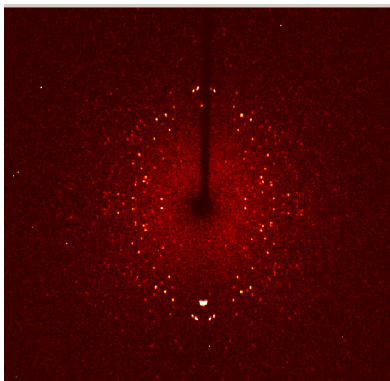


Figure 5.26 - A 360° phi scan on a small crystal. The diffraction power of the crystal is small, but with slower scans this is clearly a reasonable candidate for data collection.

6. Data Collection

The data collection process is carried out on the client computer using APEX2. Once data collection is started, exit APEX2 (optional). Data collection will continue regardless.

6.1 Start a New Project and Describe the Sample

1. In APEX2, left-click on File > New.
2. In the window that appears, enter the sample name.

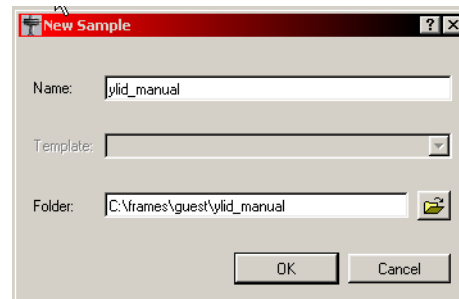
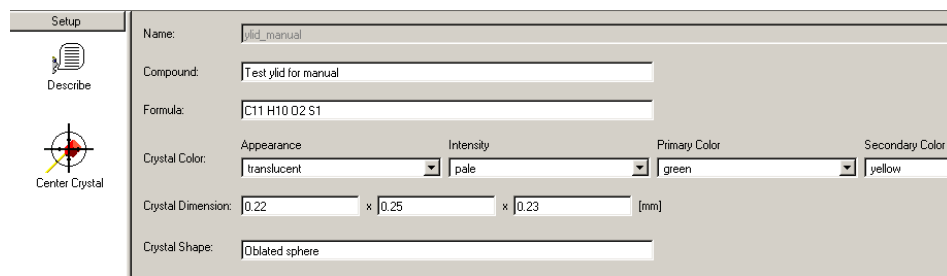


Figure 6.1 - The New Sample window

3. Click OK.
4. The task bar will appear with the Setup section open. Left-click on Describe.

5. Enter the requested information into the Describe window.



The 'Describe' window is shown with the following data:

Field	Value
Name	ylid_manual
Compound	Test ylid for manual
Formula	C11 H10 O2 S1
Crystal Color	translucent
Appearance	pale
Intensity	green
Primary Color	yellow
Secondary Color	
Crystal Dimension	0.22 x 0.25 x 0.23 [mm]
Crystal Shape	Oblated sphere

Figure 6.2 - Describe window

6. Close this module. The data will automatically save to the database.

6.2 Determine the Unit Cell

6.2.1 Collect Images

1. In the task bar, left-click on Collect and then Experiment.

If there was no connection to the instrument when this module was started, the program will either automatically connect or it will recognize that it needs to connect in order to collect images, and will ask to connect (see Figure 6.3).

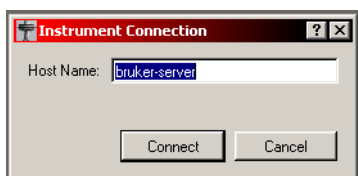


Figure 6.3 - Instrument Connection window

2. Click on Connect.
3. Click on Append Matrix Strategy at the bottom left of the window.

	Operation	Active	Distance [mm]	2Theta [deg]	Omega [deg]	Phi [deg]	Chi [deg]	Time [sec]	Width [deg]	Sweep [deg]	Direction
1	Phi Scan	Yes	40.000	30.000	-45.000	0.000	54.740 default	default	default	6.000 positive	
2	Phi Scan	Yes	40.000	30.000	-45.000	120.000	54.740 default	default	default	6.000 positive	
3	Phi Scan	Yes	40.000	30.000	-45.000	240.000	54.740 default	default	default	6.000 positive	
4	No Operation	Yes									
5	No Operation	Yes									
6	No Operation	Yes									
7	No Operation	Yes									
8	No Operation	Yes									
9	No Operation	Yes									
10	No Operation	Yes									
11	No Operation	Yes									
12	No Operation	Yes									
13	No Operation	Yes									
14	No Operation	Yes									
15	No Operation	Yes									
16	No Operation	Yes									
17	No Operation	Yes									

Figure 6.4 - Append matrix runs

- Adjust the scan time and scan width if desired. The default values are usually good. The default time of 10 seconds works for most samples, but shorter times will not adversely affect most experiments.
- Click on Execute. The view will shift to the Monitor Experiment view (see Figure 6.5). The program will collect a series of three runs with twelve frames per run. This typically takes less than ten minutes. The images will stop changing when the experiment is done. It is not necessary to wait for all runs to complete before proceeding to the harvesting step (step 6.2.2).

NOTE: Adjust the time (upper right) to match the scattering ability of the crystal (i.e., shorter exposure times for strong diffractors and longer times for weak diffractors). If the exposure times are five seconds or less, click on the check mark by Correlate Frames to turn off this feature. Frame correlation takes two exposures for each frame, each typically having half the duration of the full exposure, and then combines the two together. This is usually not necessary with shorter exposure times.

NOTE: The format for frame names is shown in Figure 6.5. APEX2 assigns every frame a name. For this figure, the name is ylid_manual_01_005.sfrm. This means that the frame is for the project ylid_manual and that this is the fifth image of the first run.

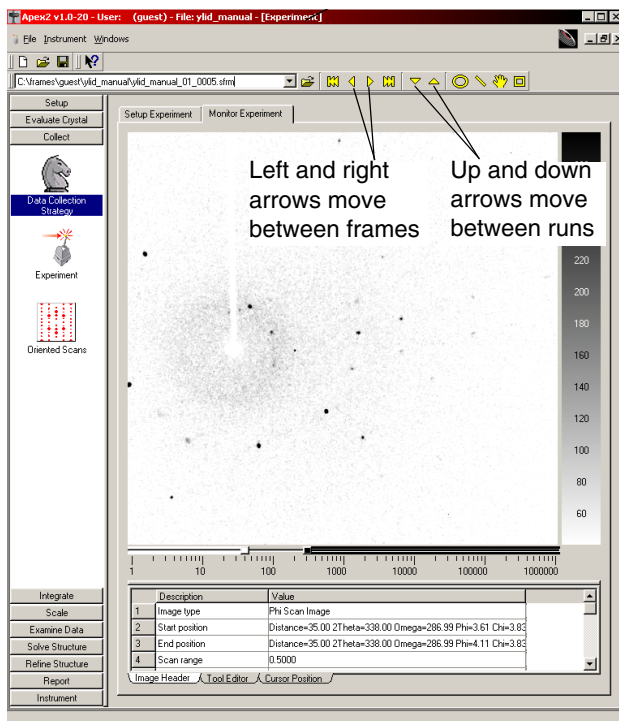


Figure 6.5 - Monitor Experiment view

To change the color of the image display (e.g., Black On White), right-click in the intensity bar to the right of the image display (see Figure 6.6).

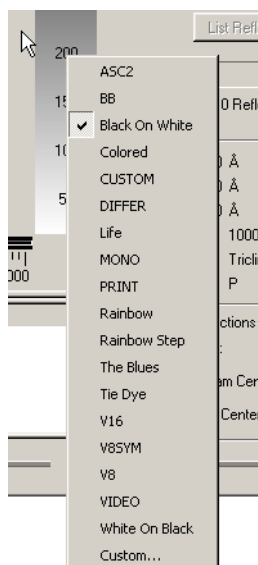


Figure 6.6 - Color tool

NOTE: After the first run is completed, there is usually sufficient information to start the indexing step.

6.2.2 Harvest the Reflections

1. Left-click on Evaluate Crystal > Determine Unit Cell.



Figure 6.7 - The Determine Unit Cell (Indexing) icon

This will open the image viewer, but with a tool bar to the right for indexing (see Figure 6.8). The plug-in initializes with the first run (e.g., matrix_01).

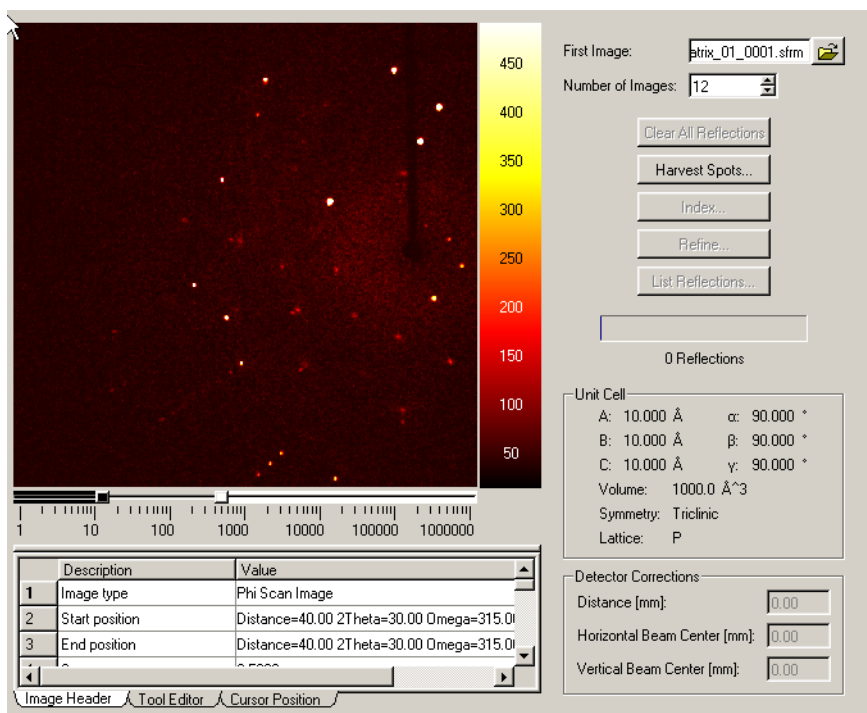


Figure 6.8 - Image viewer with indexing tool

2. The name of the first image is already entered. Click on Harvest Spots.

NOTE: All other options are gray at this point because no reflections are available.

A blue progress bar will appear as the software determines the best background level to use for harvesting. Then a window with two sliders will appear.

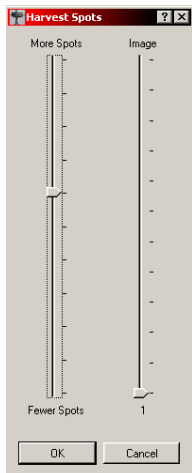


Figure 6.9 - Indexing sliders

The right slider selects which image is displayed. The left slider increases or decreases the $I/s(I)$, the cutoff criteria for accepting reflections. Generally, the defaults are acceptable.

3. Change the run number in the “First Image” box to matrix_02_0001 and click on Harvest Spots. The run number is 02. The image or frame number is 0001.
4. Change the run number in the “First Image” box to matrix_03_0001 and click on Harvest Spots.

At this point you should have 100 to 300 reflections harvested.

NOTE: If you have started harvesting before all of the matrix runs were collected, a window may pop up that says “Do you want to continue with the images that could be read?” If this happens and only one or two frames are needed to complete the run, wait, and then process the entire run. However, if you have a hundred or more spots and there are several frames yet to be collected, you can skip step 3 or 4 and go to Section 6.2.3. Then return to Section 6.2.2 and harvest the spots before refining.

6.2.3 Index the Reflections

1. Click on Index in the tool bar to the right of the image viewer. A window will open.

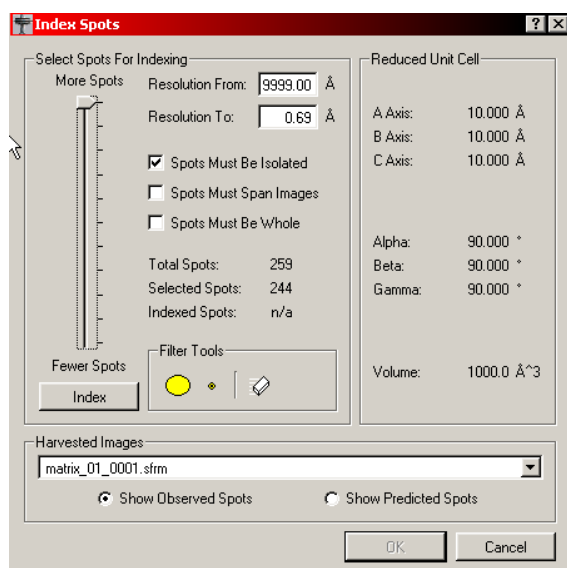


Figure 6.10 - Indexing tool

The defaults are usually acceptable. Use the slider to omit reflections with lower I/σ from the calculations. If indexing is difficult, try reducing the number of reflections used.

If indexing is difficult, use the RLATT tool. This tool is described in Appendix A.

2. Click on Index.

After approximately 30 seconds, the Index window will display a possible cell and the OK button will no longer be gray. The values shown in Figure 6.11 are reasonable for the YLID crystal.

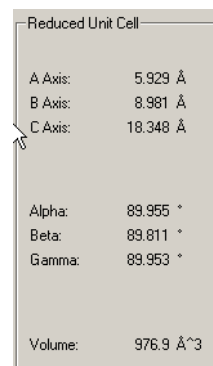


Figure 6.11 - The unit cell

The spot statistics are also acceptable with 98% (i.e., $(238/244) \times 100$) of the selected spots indexed.

Total Spots:	259
Selected Spots:	244
Indexed Spots:	238

Figure 6.12 - Focus on the spot results

There are often a few reflections that are not indexed. You can use the reciprocal lattice viewer to look at the spots used in the indexing, but refine this cell first.

3. Click on OK to accept the indexing results.

6.2.4 Refine the Unit Cell

There is not a correct order for the following steps. Use this procedure as a guideline with the main goal of creating a stable converged refinement.

1. Click Refine in the Indexing Tools menu.
The Refine Unit Cell window will open.

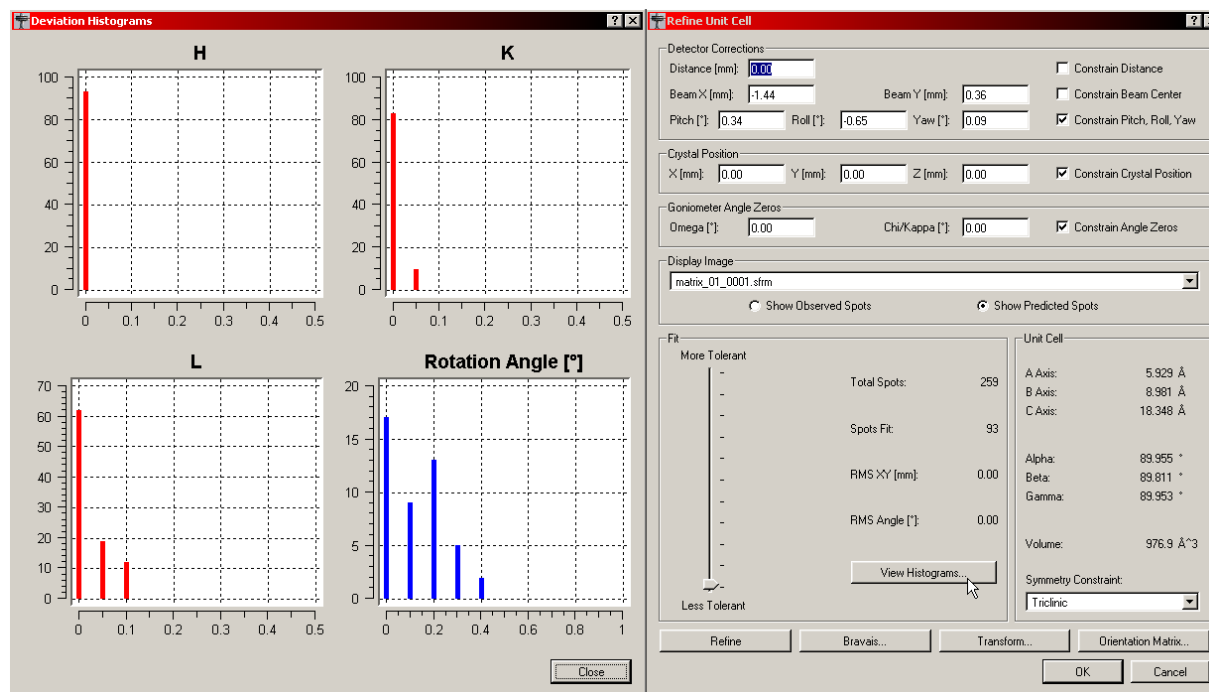


Figure 6.13 - The Refine menu with histograms displayed

The YLID test crystal should have an orthorhombic primitive cell with approximate cell dimensions of $a=5.95\text{\AA}$, $b=9.03\text{\AA}$, $c=18.38\text{\AA}$, and $\alpha=\beta=\gamma=90^\circ$.

2. Click View Histograms. The histograms show how observed data compares to the data calculated using the current unit cell. The HKL values should be close to integers and the rotation angle differences should not be significantly larger than the step size used to collect the matrix frames.
3. Click Refine several times.
4. Check the Constrain Distance and Constrain Beam Center boxes.

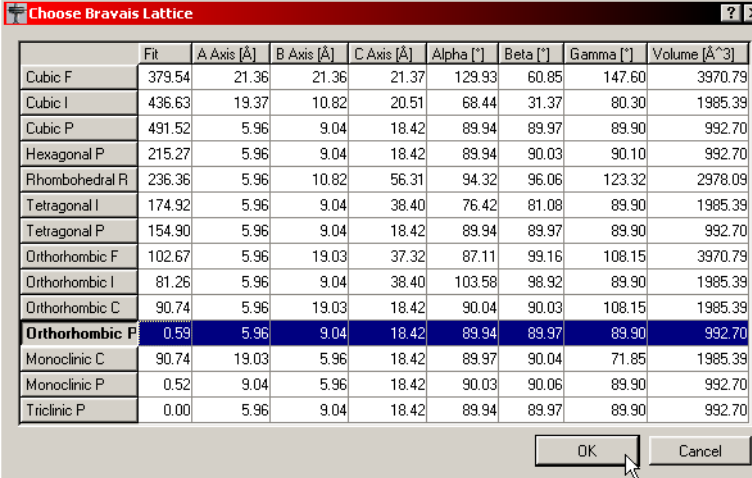
NOTE: Check the constraints to fix the parameters listed. Uncheck the constraints to allow the parameters to refine.

5. Uncheck Constrain Pitch, Roll, and Yaw.
6. Click Refine several times.
7. Uncheck Constrain Distance and Constrain Beam Center, and check Constrain Pitch, Roll, and Yaw.
8. Click Refine several times.

NOTE: In most cases, the angle zeroes are close to zero and should not be refined. The crystal should now be aligned, so refinement of the crystal center is not necessary.

6.2.5 Determine the Bravais Lattice

1. After refining, click Bravais Lattice and look for other unit cell choices (i.e., look at fit values).



	Fit	A Axis [Å]	B Axis [Å]	C Axis [Å]	Alpha [°]	Beta [°]	Gamma [°]	Volume [Å³]
Cubic F	379.54	21.36	21.36	21.37	129.93	60.85	147.60	3970.79
Cubic I	436.63	19.37	10.82	20.51	68.44	31.37	80.30	1985.39
Cubic P	491.52	5.96	9.04	18.42	89.94	89.97	89.90	992.70
Hexagonal P	215.27	5.96	9.04	18.42	89.94	90.03	90.10	992.70
Rhombohedral R	236.36	5.96	10.82	56.31	94.32	96.06	123.32	2978.09
Tetragonal I	174.92	5.96	9.04	38.40	76.42	81.08	89.90	1985.39
Tetragonal P	154.90	5.96	9.04	18.42	89.94	89.97	89.90	992.70
Orthorhombic F	102.67	5.96	19.03	37.32	87.11	99.16	108.15	3970.79
Orthorhombic I	81.26	5.96	9.04	38.40	103.58	98.92	89.90	1985.39
Orthorhombic C	90.74	5.96	19.03	18.42	90.04	90.03	108.15	1985.39
Orthorhombic P	0.59	5.96	9.04	18.42	89.94	89.97	89.90	992.70
Monoclinic C	90.74	19.03	5.96	18.42	89.97	90.04	71.85	1985.39
Monoclinic P	0.52	9.04	5.96	18.42	90.03	90.06	89.90	992.70
Triclinic P	0.00	5.96	9.04	18.42	89.94	89.97	89.90	992.70

Figure 6.14 - Bravais lattice display

Note that even though monoclinic has a slightly better fit, the software makes the correct choice of the higher symmetry cell.

Now you have a unit cell ready for determining a data collection strategy.

2. Click on the appropriate Bravais lattice (in this case, Orthorhombic).
3. Press OK to accept the suggested lattice settings.
4. Refine again.
5. Refine for several more cycles, changing the constraints one or two at a time.

6.3 Determine the Data Collection Strategy

APEX2 includes a powerful algorithm, COSMO, for determining an efficient strategy that fully utilizes the flexibility of your instrument.

1. Left-click on Collect > Data Collection Strategy.

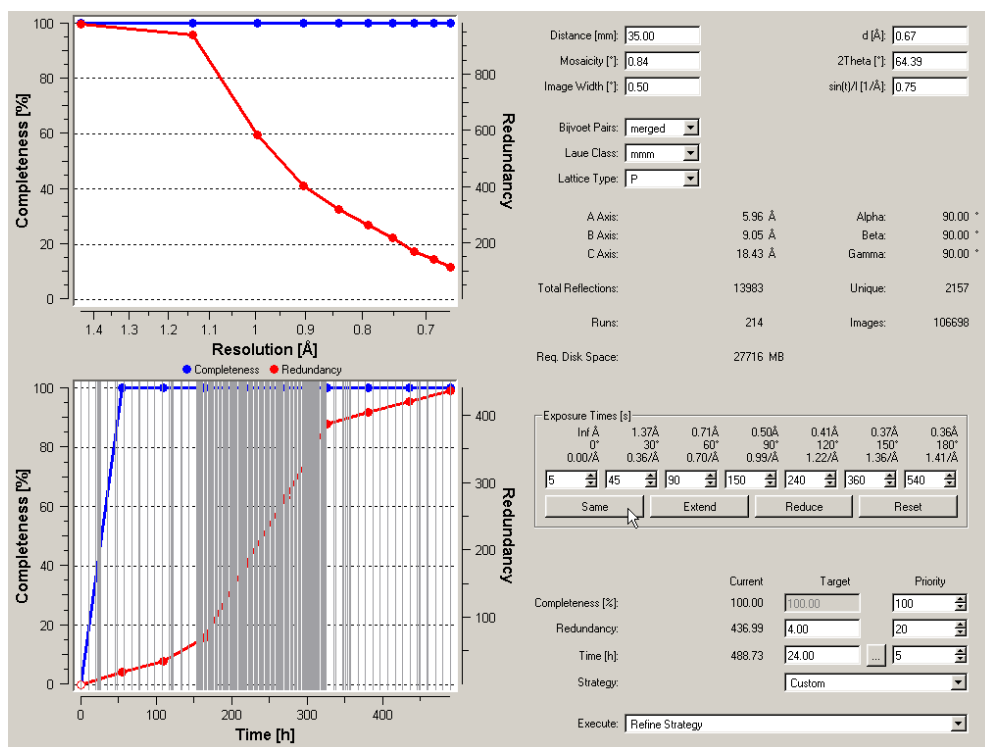


Figure 6.15 - The strategy display

NOTE: COSMO will use information from cell determination to set defaults. You can modify the suggested values.

2. Check the inputs for defining the data collection.
 - 2.1 Set the data collection distance. For SMART APEX II systems, this should be set to the actual detector distance. For Kappa APEX II systems, there is a variable (DX) and the distance will default to the shortest reasonable distance. For the APEX II detector, the distance in millimeters should generally be about the same as the longest cell dimension in angstroms. Typically, distances ranging from 35 to 45 are reasonable.
 - 2.2 Set the exposure time and press Enter. For normal crystals on an APEX II, five seconds is a reasonable time.
 - 2.3 Click Same to set all of the times to be the same.
-

NOTE: If the “Same” feature is not chosen, the times for shells can be set to collect high angle data more slowly than inner shell data.

- 2.4 Set the desired resolution (0.75 is a reasonable value).
- 2.5 Check the other values (Laue class, Lattice, etc.).
- 2.6 Each time a value is changed, COSMO recalculates the statistics for the runs. The results are displayed in the column labeled Current.
- 2.7 Below the Target and Priority columns is a pull-down menu with several different strategies. Choose the one that best meets the needs of the experiment (e.g., “Best in 2 hours” for the example used here).

At this point, if all of the runs available were collected it would take 183.98 hours and have a redundancy of 452.86. Clearly this is not desirable.

3. Click on Refine Strategy.
4. A list of options will appear. Click on Refine Strategy again.



Figure 6.16 - Click on Refine Strategy

NOTE: The objective in Refine Strategy (COSMO) is to get good completion (98% or better) with high redundancy in a reasonable amount of time. When COSMO is first started it will tell you the completion, redundancy, and time for all of the available runs. It is almost never necessary to let COSMO run to completion. Typically, it should be stopped when completion is greater than 99% and the time is close to what is desired.

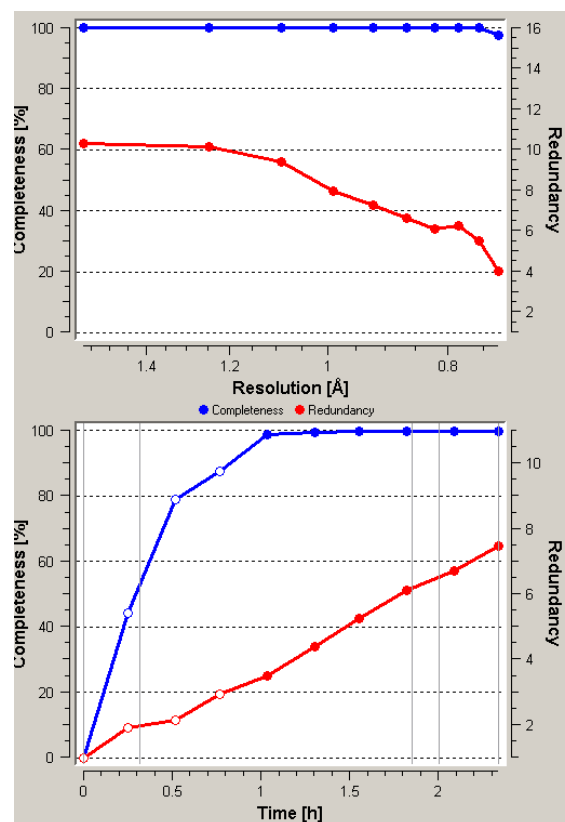


Figure 6.17 - Completeness and Redundancy chart

In this example, as shown in Figure 6.17, the completion is 99.76% and the time is approximately 2.33 hours.

NOTE: Time estimates are approximate. They depend on the number of rescans, general instrument overhead, backlash compensation, etc. If estimated times are consistently longer or shorter, modify the COSMO hardware profile.

- Click Stop when the completeness nears 100% and the time and redundancy approach the desired values. It is not necessary to wait until the refinement reaches 100%.

Current	Target	Priority
99.76	100.00	100
7.48	50.00	5
2.33	2.00	10

Best in 2 hours

Figure 6.18 - Strategy Status and Priority control

- Click Refine Strategy.
- A list of options will appear. Click on Sort Runs for Completeness.

Execute:

- Refine Strategy
- Refine Strategy
- Sort Runs for Completeness

Figure 6.19 - Choose "Sort Runs for Completeness"

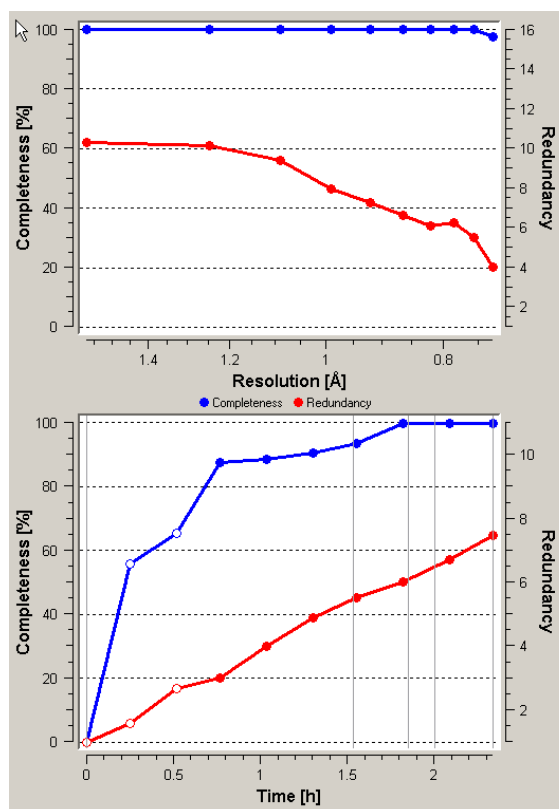


Figure 6.20 - Completeness and Redundancy charts after sorting for completeness

8. To look at the actual runs chosen, go to View > Detailed Strategy.

This will open a window that shows the runs to be collected (see Figure 6.21).



	Scan Type	CCD	2Theta	Omega	Phi	Chi	Axis	Width	#Images	Sweep	Time
1	Strategy	35.00	-22.00	+286.99	-358.39	+3.83	Phi		739	369.50	7.5
2	Strategy	35.00	-32.00	-24.79	+27.75	-99.47	Omega		153	76.50	7.5
3	Strategy	35.00	-7.00	-4.01	-26.26	-55.59	Omega		74	37.00	7.5
4	Strategy	35.00	+28.00	+48.51	-14.91	-34.09	Phi		162	81.00	7.5
5	Strategy	35.00	-32.00	+317.27	+63.87	+93.86	Phi		127	63.50	7.5
6	Strategy	35.00	-32.00	-170.70	+23.43	+98.83	Omega	+0.50	96	48.00	7.5

Figure 6.21 - Runs to be collected

NOTE: If for some reason it is necessary to start over, change the distance slightly (by 0.02 for example) and press Return. COSMO will reload all of the possible runs.

You are now ready to collect data.

6.4 Data Collection/Run Experiment

1. Click Collect > Experiment.
2. Go back to the experiment window and delete the three matrix runs if they are still there.

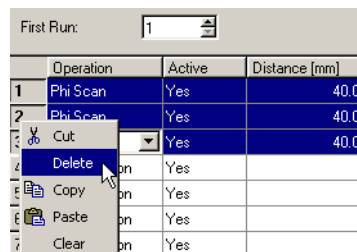


Figure 6.22 - Deleting the matrix runs

3. Click Append Strategy.

4. The program changes the name to the name of the current project (in this example, ylid_manual).

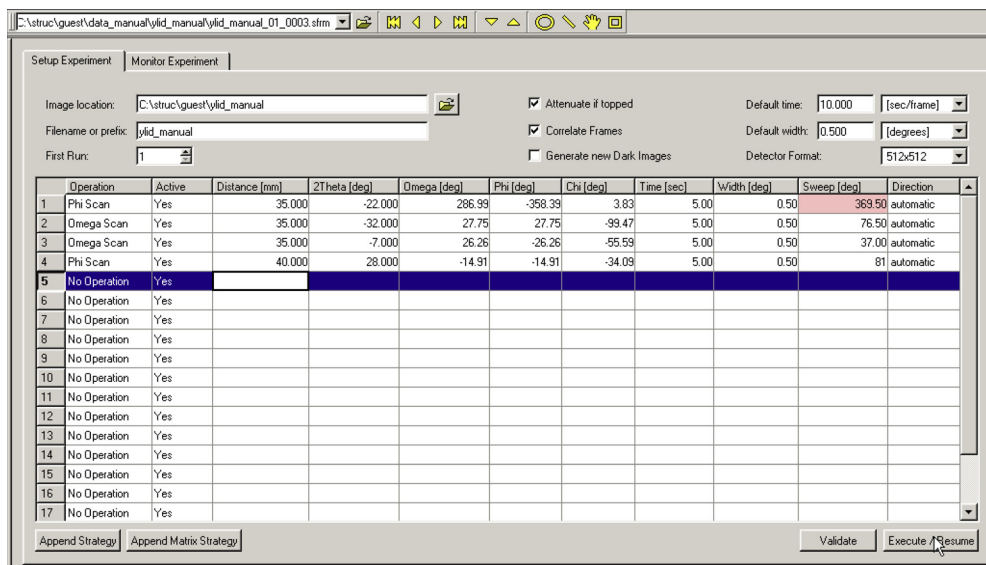


Figure 6.23 - Experiment view with strategy appended. In Version 1.26 and later, the Execute and Resume buttons are separated. Execute will force the collection of all data. Resume will start at the point where the data collection was interrupted.

NOTE: At the top of the experiment window are controls for data collection. Usually, the default values are correct. For data collection times of less than five seconds, correlation can usually be turned off. If new dark frames are required, APEX2 will automatically collect them. Checking “Generate New Darks” will force the collection of darks before every run. In Figure 6.23, the time and width are explicitly set for each run, so changing the default width and time will have no effect. If the explicit time or width for a run is deleted so that the box is empty, the word “default” appears and the default values at the top right will be used.

5. Click Execute/Resume. The focus will shift to Monitor Experiment and images will start to appear. This may take a minute or two if new darks are being collected.

NOTE: When resuming after a power failure, APEX2 will automatically skip images that were previously collected with matching angles and generator settings. Otherwise, it will ask if you want to overwrite the images.

NOTE: After data collection is started, the experiment window can be closed and APEX2 can be stopped. The server computer must be left on. If communications are lost between the client and the server, frame data is stored on the server. Typically they will be in the directory C:\frames\. They should be copied to the correct project directory before starting integration.

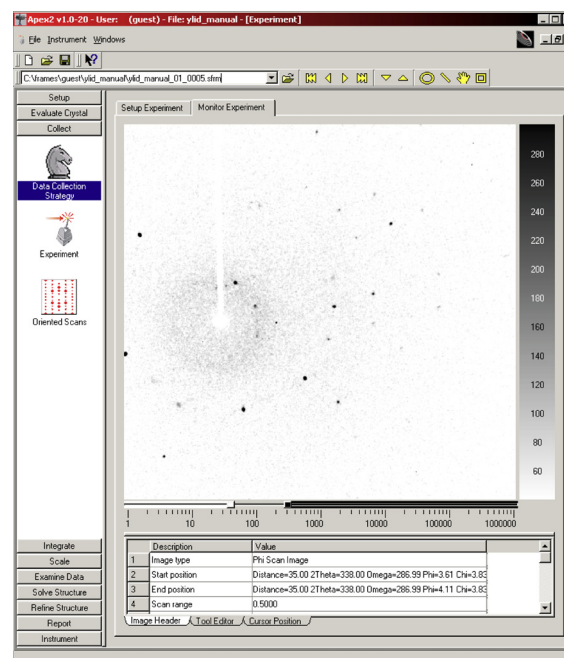


Figure 6.24 - Monitor Experiment view

7. Data Integration and Scaling

Before the data can be used to solve and refine the crystal structure, it is necessary to convert the information recorded on the frames into a set of integrated intensities, and to scale all of the data.

7.1 Integration

1. Click on Integrate in the task bar.
2. Click on the Integrate Images icon. The following window will open.

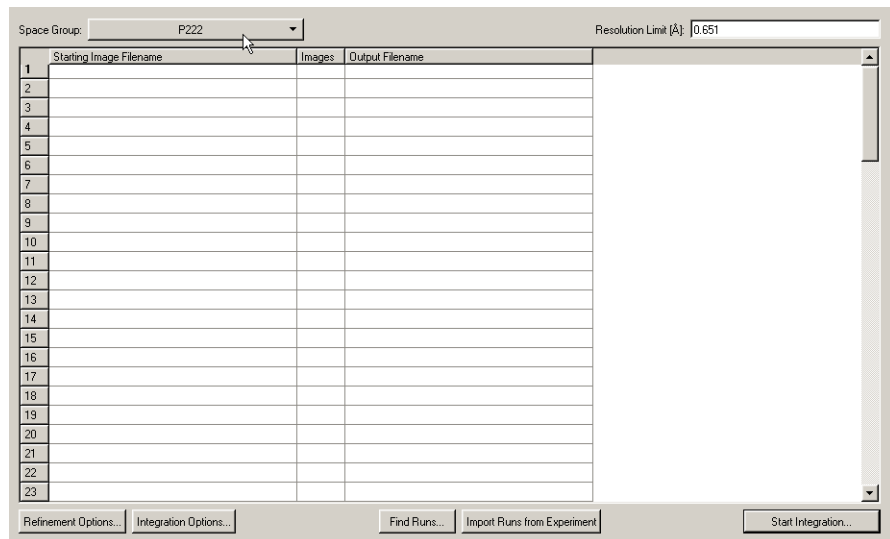


Figure 7.1 - Initial integration window

3. Check the default values.

There are two items of interest at the top of the window: the Space Group tool and the Resolution Limit value.



Figure 7.2 - The Space Group tool

The Space Group tool allows the user to set the symmetry for integration. Typically, this value is correct when the Integration window opens.

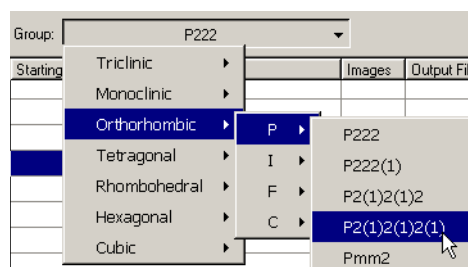


Figure 7.3 - Choosing P2(1)2(1)2(1) with the Space Group tool

The Resolution window on the upper right sets the upper limit of resolution for integration.



Figure 7.4 - The Resolution tool

The APEX2 Suite will suggest a resolution cut-off. In this case, the suggested value has been changed to 0.75.

At the bottom of the window are two buttons for defining the data collection runs to be integrated.

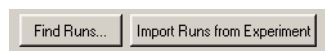


Figure 7.5 - The Find and Import Runs buttons

The Import Runs button determines the runs to be integrated from the experiment that has just been submitted. Using this button allows the user to start integration while the data is still being collected.

NOTE: SAINT will integrate all of the data currently measured and then will wait for the next image, integrate that, wait for the next, etc.

The Find Runs button is used to browse to the set of runs to be integrated. This button is normally used when the data collection is finished.

- 1. Click on Find Runs.

The following window will open. Usually it will have the correct directory and base name as the defaults. If these are not correct, then use the Browse button to find the runs for the data collection of interest.

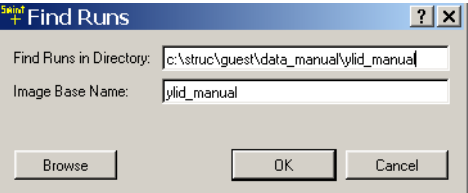


Figure 7.6 - Find Runs tool view

Both the Import Runs from Experiment and the Find Runs buttons will generate a list of runs as shown in Figure 7.7.

Space Group: P222			
	Starting Image Filename	Images	Output Filename
1	C:\struc\guest\data_manual\yolid_manual\yolid_manua	739	\structures\data_for_manual\yolid_manual\work\yolid_n
2	C:\struc\guest\data_manual\yolid_manual\yolid_manua	153	\structures\data_for_manual\yolid_manual\work\yolid_n
3	C:\struc\guest\data_manual\yolid_manual\yolid_manua	74	\structures\data_for_manual\yolid_manual\work\yolid_n
4	C:\struc\guest\data_manual\yolid_manual\yolid_manua	161	\structures\data_for_manual\yolid_manual\work\yolid_n

Figure 7.7 - Completed runs list

NOTE: To integrate runs from a CD or DVD, browse to the device and find the runs to be integrated. APEX2 will automatically write the result in the work directory for the current project.

Figure 7.8 shows a run list entry being modified. (A) shows the original run information. (B) shows the run with the starting image number changed to 51. (C) shows the number of frames to process changed to 111.

Double-click on any of the fields in this list to open up that value for editing.

A	C:\struc\guest\data_manual\ylid_manual\ylid_manua	74
	manual\ylid_manual\ylid_manual_04_0001.sfm	161
B	C:\struc\guest\data_manual\ylid_manual\ylid_manua	74
	manual\ylid_manual\ylid_manual_04_0051.sfm	161
C	C:\struc\guest\data_manual\ylid_manual\ylid_manua	74
	C:\struc\guest\data_manual\ylid_manual\ylid_manua	111

Figure 7.8 - Example of manually editing a run list

2. Check the refinement defaults.

The two buttons on the bottom far left are used to change the default options for the refinement of the data.

The default values are generally very good. There is seldom any need to change those in the Refine window.

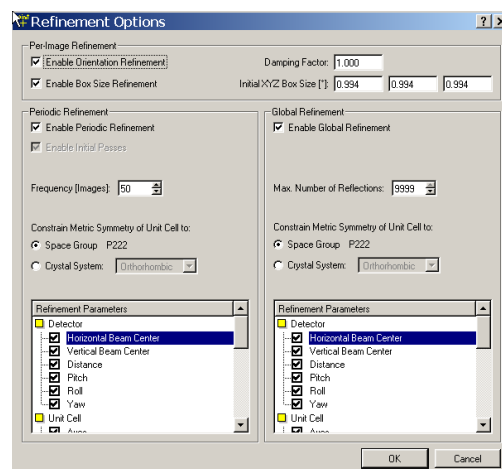


Figure 7.9 - Refine Window

3. Change the default options for the integration in the “Integration Options” window.

The Integration Options window has a few values that are changed more frequently.

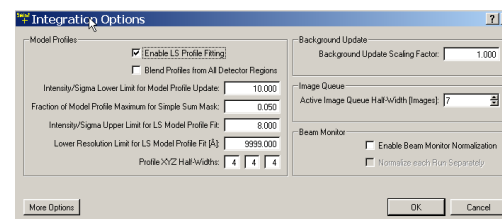


Figure 7.10 - Integration Options window

The default values in the Model Profiles window are usually good.

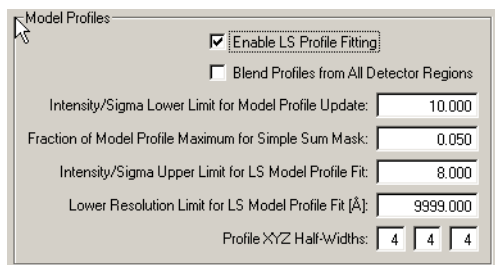


Figure 7.11 - Model Profiles window

For weakly-diffracting crystals it is sometimes helpful to change some options in the Model Profiles window. Reducing the Intensity/Sigma ratios from 10 and 9 to, for example, 5 and 4 or 3 gives the profile fitting algorithm more reflections to model. Increasing the profile blending by clicking on “Blend Profiles from All Detector Regions” can also help with weakly-diffracting crystals.

4. Click on More Options. This results in an expanded window (see Figure 7.12) and gives access to several other useful features, in particular Active Mask (see Figure 7.13), Algorithm (see Figure 7.14), and Image Queue (see Figure 7.15).

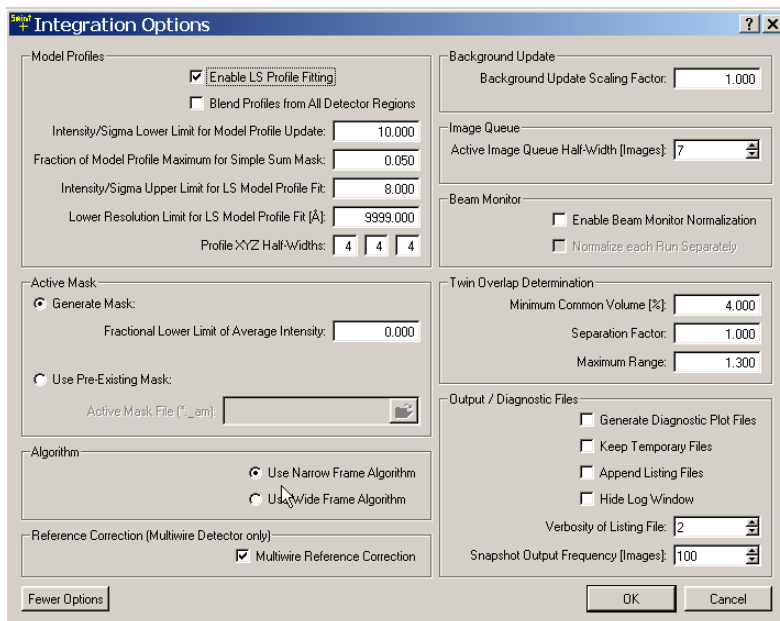


Figure 7.12 - The Integration Options window with "More Options" selected

7.1.1 Active Mask

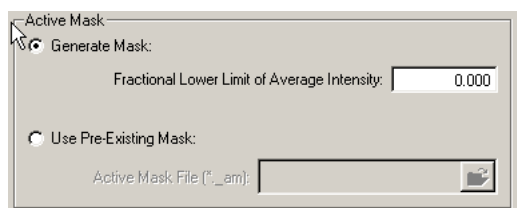


Figure 7.13 - The Active Mask tool

The Active Mask is used to mask out areas that are covered by the beamstop, low-temp nozzle, or other obstructions. Typically a value of 0.7 is good here. SAINT will determine an active mask for each run.

NOTE: If this option is set, the user should examine the active mask images that are written into the work directory with the name `basename_am_##.sfrm`. There is one for each run. These images can be opened with the image viewer. They should be blank except for an area in a different color showing the shape of the obstruction, typically the beamstop.

7.1.2 Algorithm



Figure 7.14 - The Narrow/Wide Algorithm tool

The algorithm buttons are used to change from narrow to wide frame integration. Frames collected with narrow scan widths (less than one degree) should be integrated as narrow frames. Those greater than one degree should be integrated as wide frames.

7.1.3 Image Queue

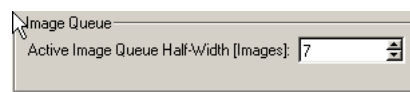


Figure 7.15 - The Image Queue tool

The Image Queue sets the number of frames being used to determine profiles. For crystals with high mosaic spread (large, wide reflections), increasing the queue size can improve integration.

- Click on the Start Integration button on the far right of the bottom row. This will start the integration and will open the SaintChart window for monitoring the integration.
- Examine the output. Double-click on any of the output entries for one additional tool—the ls button at the far right of the line (see Figure 7.16). This will open the SAINT listing file for that integration run.



Figure 7.16 - Using the runs list ls tool

7.2 SaintChart

SaintChart is a powerful tool for monitoring the progress of the calculation and for graphically presenting the results of the integration process.

7.2.1 Monitor the Progress of the Integration Run

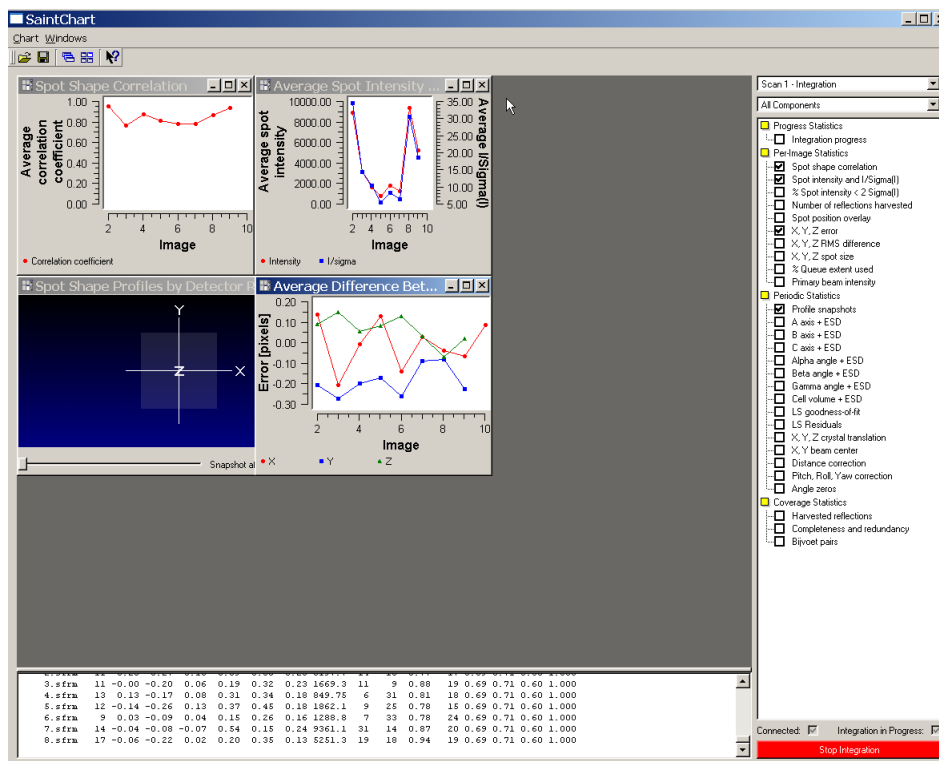


Figure 7.17 - SaintChart view

As shown in Figure 7.17, when the SaintChart window opens the graphs are small, and under Linux the text area is large.

If desired, change the view.

Change the text area by clicking and dragging on the line between the text and image areas.

Expand the windows to fill the available image area by clicking on the four-square icon  in the upper left (see Figure 7.18).

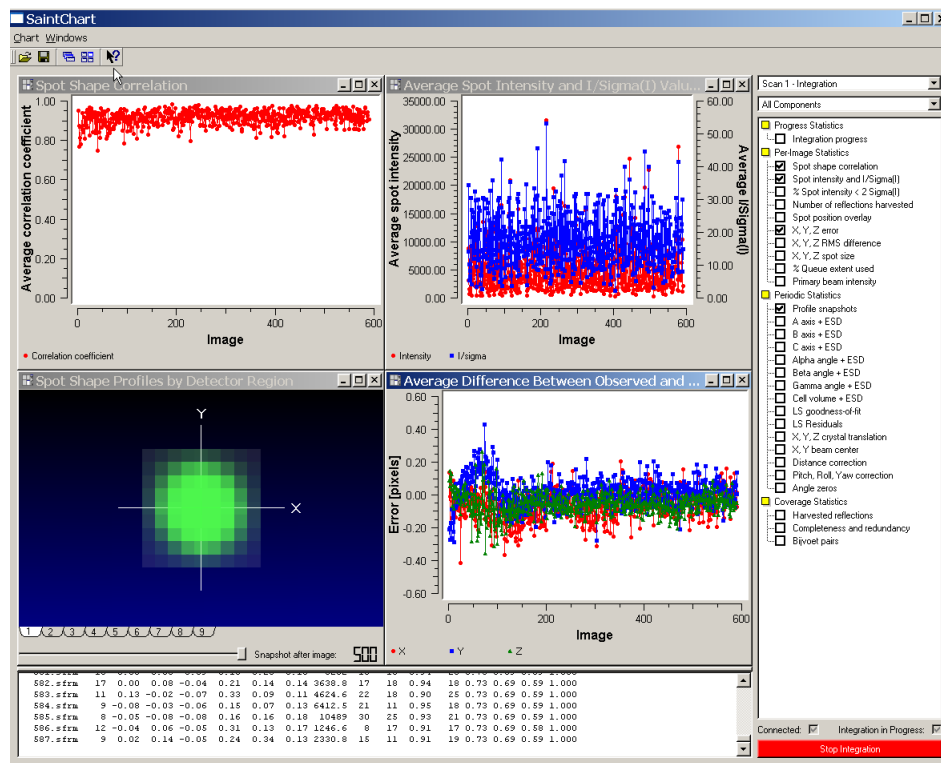
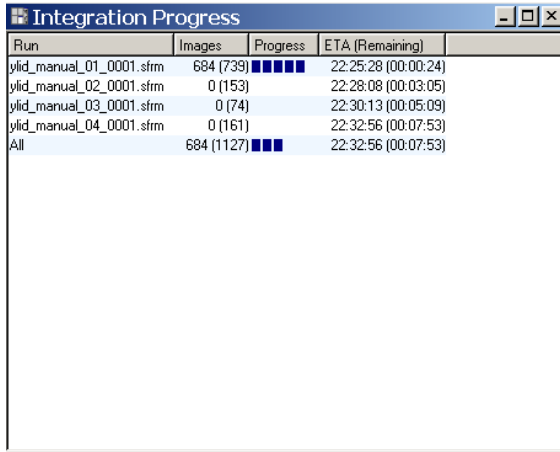


Figure 7.18 - SaintChart view with graphical area expanded

To the right of the image area are a series of check boxes that select the displays.

1. Click on the check boxes to add or remove displays. Click on Integration Progress to add the new display seen in Figure 7.19. When the integration is complete, the blue progress bars will be full and the remaining time will be zero.



The image shows a window titled "Integration Progress" with a table containing the following data:

Run	Images	Progress	ETA (Remaining)
ylid_manual_01_0001.sfrm	684 (739)	■■■■■	22:25:28 (00:00:24)
ylid_manual_02_0001.sfrm	0 (153)		22:28:08 (00:03:05)
ylid_manual_03_0001.sfrm	0 (74)		22:30:13 (00:05:09)
ylid_manual_04_0001.sfrm	0 (161)		22:32:56 (00:07:53)
All	684 (1127)	■■■	22:32:56 (00:07:53)

Figure 7.19 - Integration Progress display

- Click on Spot Position Overlay (in Per-Image Statistics) to add one of the most powerful windows for monitoring the progress of the integration. Pull up on the Window Tool Bar for Integrated Spots (the cursor arrow is pointing to this tool bar in Figure 7.20) to display the entire window (see Figure 7.21).

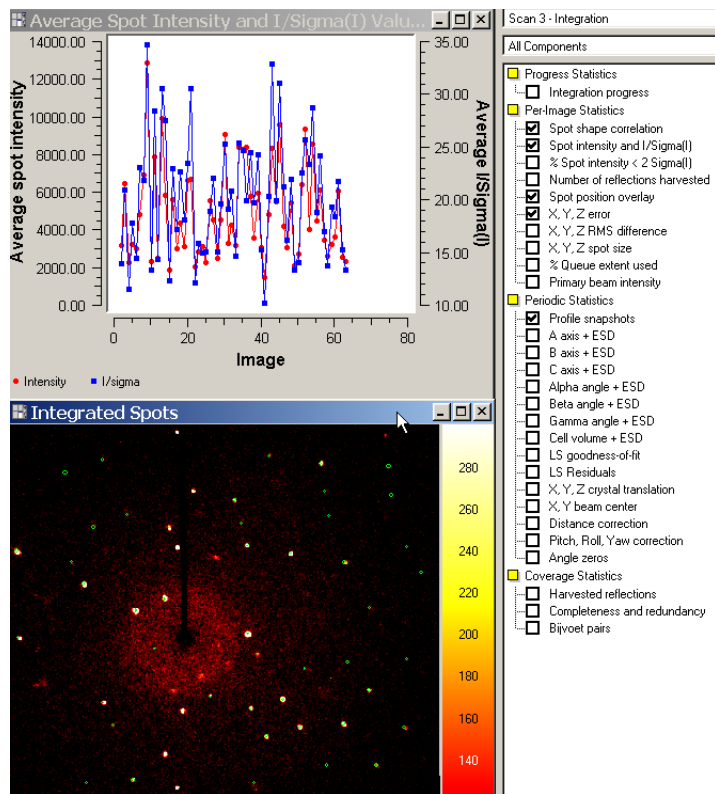


Figure 7.20 - Spot Position Overlay

In this full view, the Integrated Spots window looks much like the image viewer used previously. At the bottom of the window is an entry box that can be used to change the display frequency of the Integrated Spots images. The default is to display every fifth image.

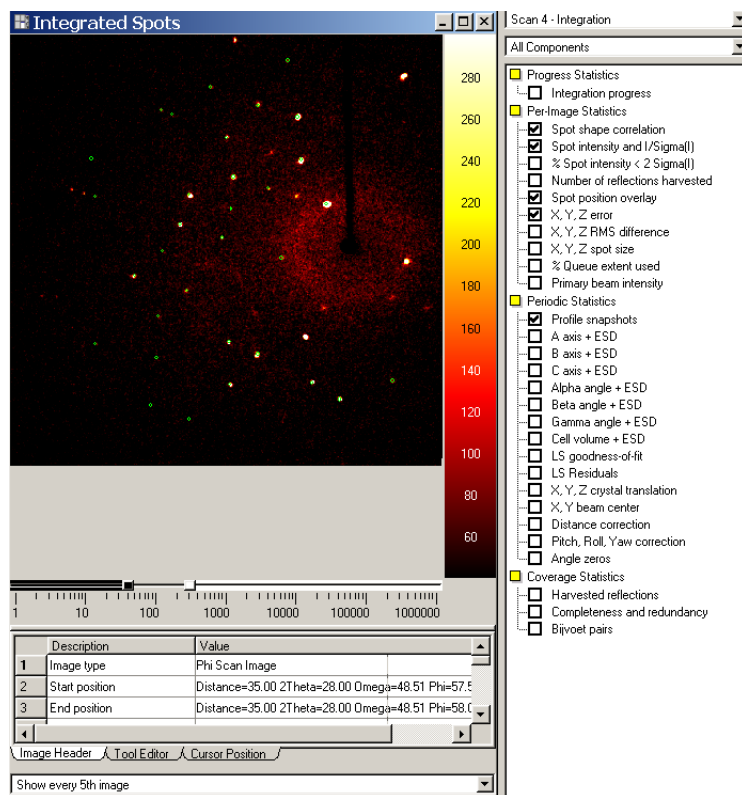


Figure 7.21 - Full view of the Spot Position Overlay

7.2.2 Examine Final Results

At the end of integration, examine this text critically. In particular, look at the Overall Rsym, the Coverage Statistics and the Unconstrained Unit Cell refinement.

1. Set up to view SAINT text output. At the bottom of the SaintChart window is a scrolling text window that displays the text output from SAINT. (This window can be expanded by dragging the horizontal bar above the

text as indicated by the position of the mouse cursor in Figure 7.22).

2. Examine the final results.

2.1 Overall Rsym and Coverage Statistics (Figure 7.22)

The overall Rsym is usually less than 8%. Higher values may indicate problems with absorption, twinning or poor crystal quality.

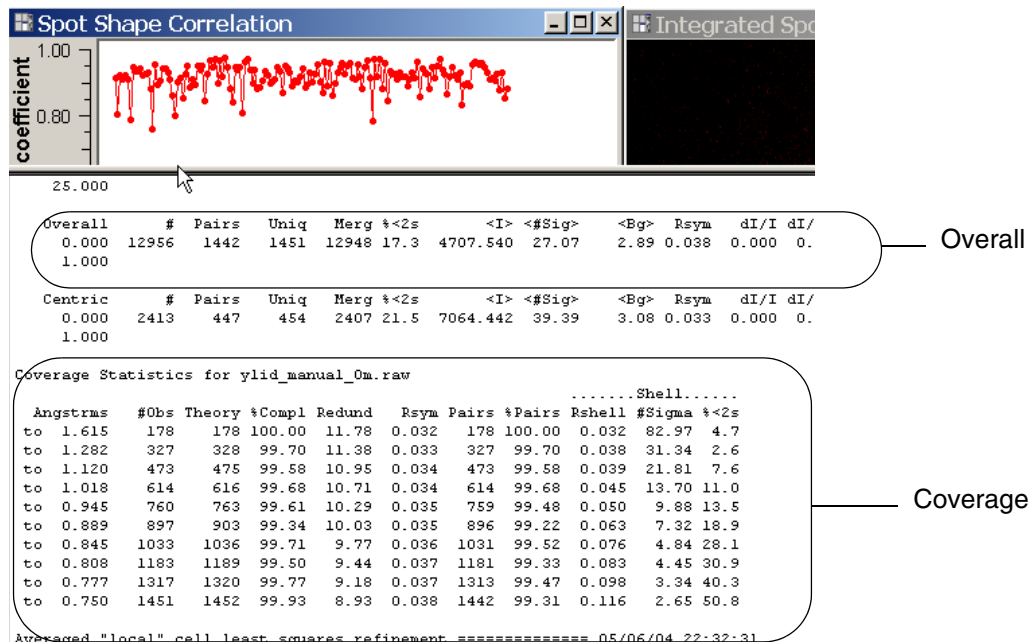


Figure 7.22 - Final SAINT Overall Rsym and Coverage Statistics

The coverage statistics are reported by resolution shells. The values in the Rsym column give the cumulative agreement of equivalent reflections. The Rshell values give agreement within a particular shell. These change faster than the Rsym values. The %<2s column is useful for checking where the weak data becomes predominant (greater than 50 or 60%). The #Sigma column gives the average I/sigma for each shell. If that number is less than 1.5 or 2, then little significant data is being measured in that shell.

2.2 Unconstrained Unit Cell Refinement (Figure 7.23)

Check the angles in the unconstrained unit cell refinement to get a quick confirmation that the initial lattice determination was correct. In the unconstrained refinement, angles that are required to be 90 or 120 are allowed to refine. If they differ considerably from the expected value, then the data should be checked carefully.

```
Unconstrained global unit cell refinement ===== 05/06/04 22:32:31

Performing final unit cell least squares on file d:\structures\data_for_manual\ylio
Input file contains      5552 reflections

Global Refinement -- single-component data...
Maximum allowed reflections =      9999
One reflection will be stored in memory per      1 reflection(s) read

Orientation least squares, component  1 (5552 input reflections)...
Wavelength, relative uncertainty:  0.7107300,  0.0000089

Reflection Summary:
'RLV.Excl' are reflections excluded after cycle 1 because RLV error exceeded 0.0250
Component      Input  RLV.Excl      Used  WorstRes   BestRes   Min.2Th   Max.2Th
          1         5552          0     5552    8.1065    0.7884    5.025    53.580

Orientation ('UB') matrix:
-0.0584509   0.0333484  -0.0483062
 0.0005328   0.1049404   0.0175150
 0.1573586   0.0121009  -0.0180036

          A           B           C       Alpha       Beta       Gamma          Vol
        5.9572       9.0273      18.3668      90.088      89.999      90.033      987.72
        0.0001       0.0002       0.0004       0.001       0.001       0.001         0.05
Corrected for goodness of fit:
        0.0005       0.0008       0.0019       0.005       0.005       0.005         0.22
```

Figure 7.23 - Unconstrained unit cell refinement

7.3 Scale

The scaling process uses the SADABS program to put all of the measured data on the same scale. This process involves five steps:

1. Set up input files.
2. Parameter refinement.
3. Error model refinement.
4. Display diagnostics.
5. Exit.

7.3.1 Set up Input Files

1. Click on Scale > Scale in the task bar. Figure 7.24 shows a portion of the initial Scale work area. The defaults and file names are typically correct.

Base: ylid_manual

Output .hkl file: ylid_manual_0m.hkl

Diagnostic Plots File Name: ylid_manuallm.eps

Title of Diagnostic Plots: ylid_manual

Log File: ylid_manuallm.abs

☒ Use only centrosymmetric point groups

Point Group: mmm

☐ Additional Spherical Absorption Correction

Mu'r of Equivalent Sphere:

☒ Lambda/2 Correction

Correction Factor: 0.0015

Allow for crystal decomposition by B-value refinement: None

Extra Linear Correction to be Applied to Each Reflection: None

Figure 7.24 - Input setup window

7.3.2 Parameter Refinement

1. Click on Refine. Figure 7.25 shows a typical refinement result. The R-values are reduced and the Mean Weight increases. Typical final values are 3–4% for the R-values and 0.95–0.98 for the Mean Weight.

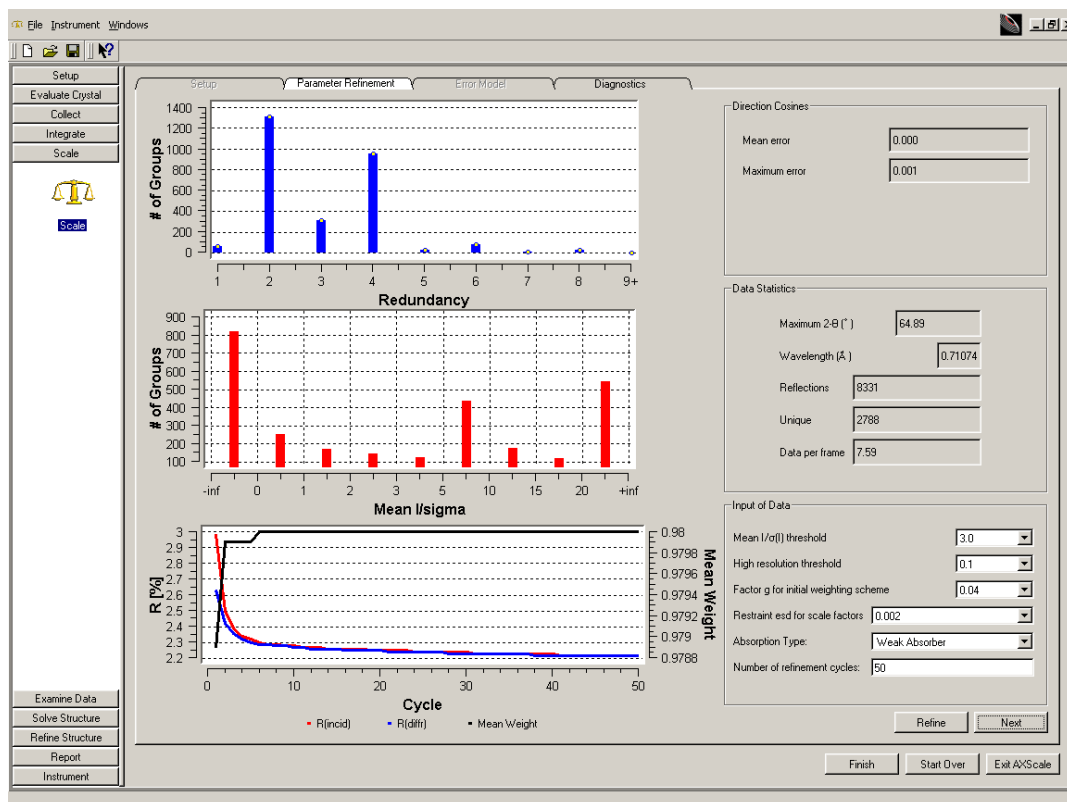


Figure 7.25 - XScale window after refinement

7.3.3 Error Model Refinement

Figure 7.26 shows typical Error Model results.

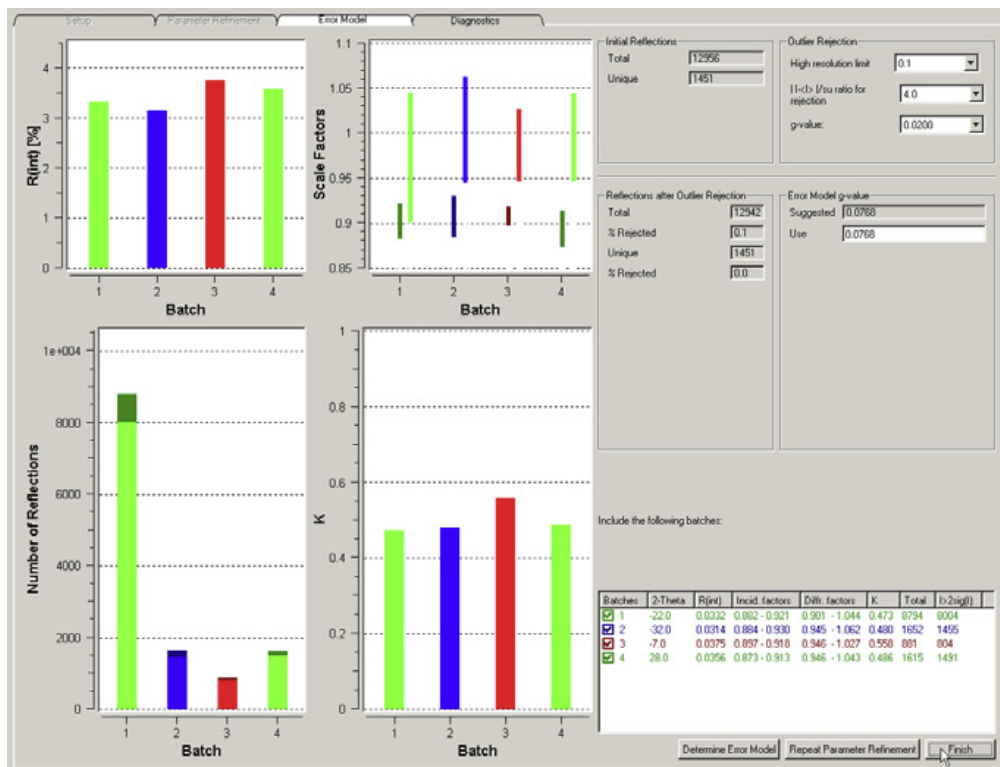


Figure 7.26 - Error Model results

If the R-values in one run are significantly higher than the others, you can repeat the entire scaling calculation with that run omitted. Click the check box by the “bad” run and then click Repeat Parameter Refinement.

7.3.4 Examine Display Diagnostics

The Display Diagnostics provide valuable insight into the quality of the data and possible problems with the data. Click on the tabs at the bottom of the screen to view diagnostic data.

1. Click Finish to produce a series of diagnostic plots. Some of these are reviews of the plots that have already been displayed.

The **Scale Variations** plot (Figure 7.27) shows the overall variation in Scale and R(int) for the individual frames. The Scale plot should be flat (for highly absorbing, irregularly shaped crystals it will probably be sinusoidal) and the R(int) plot should not show large variations (more than 2%).

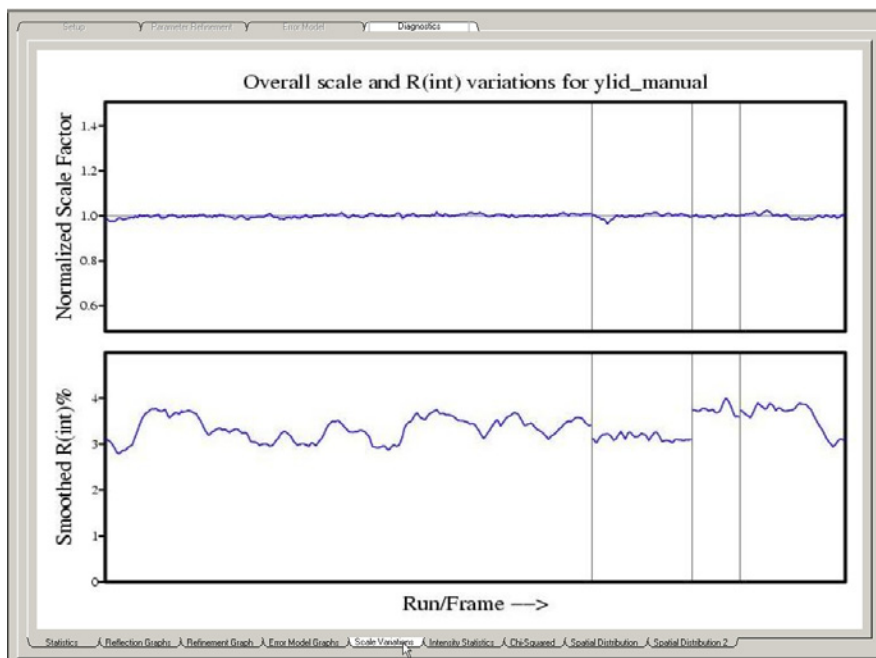


Figure 7.27 - Overall Scale and R(int) variations

The **Intensity Statistics** plots look at $R(\text{int})$ and $|E^2 - 1|$ as a function of resolution. Typical plots of $R(\text{int})$ versus resolution increase to the right as seen in Figure 7.28. An $|E^2 - 1|$ plot should have a constant resolution. $|E^2 - 1|$ is a strong indicator of centric and acentric space groups. The two horizontal lines indicate the expected values for centric (top) and acentric (bottom) space groups. This plot clearly suggests an acentric space group.

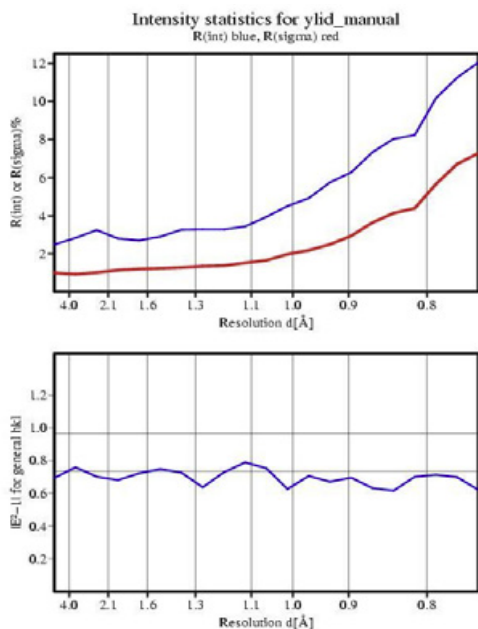


Figure 7.28 - Intensity Statistics

The plots of **Chi-Squared** values for the data as a function of resolution and intensity should be mostly flat. Values below one are not unusual for weaker high-resolution data. The plots shown in Figure 7.29 are typical.

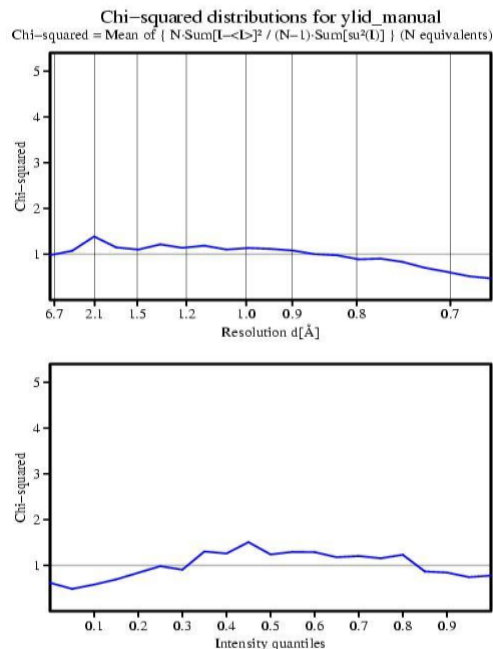


Figure 7.29 - Chi-squared

The **Spatial Distribution** plots are generated for each data collection run. They indicate, using red and blue dots, points of average intensity for a particular reflection after scaling. The actual intensity measured at that spot on the detector may vary by more than three standard deviations. Figure 7.30 shows two spatial distribution plots side by side. This is the way they are typically output by the scaling process.

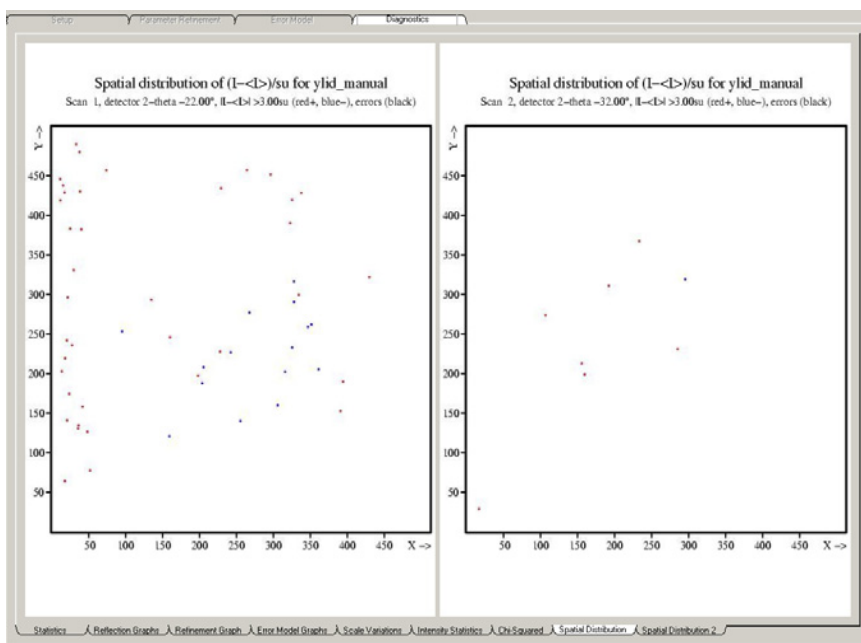


Figure 7.30 - Spatial distribution

Figure 7.31 shows a single plot so that the dots are easier to see. Note that the points of disagreement are spread fairly evenly over the entire detector face.

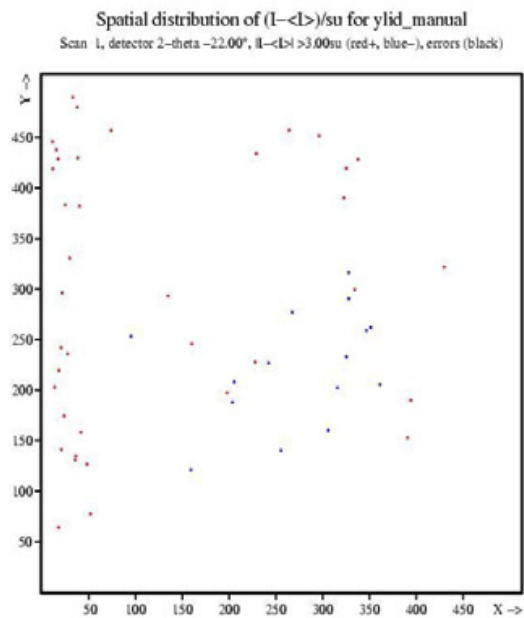


Figure 7.31 - A single Spatial Distribution

If the spots are clustered in an area or if there are significantly more spots of one color than there are of another, then the data should be examined critically. Figure 7.32 is from another data set where the Active Mask was not used during integration and the reflections collected in that area are consistently smaller than expected. This kind of problem may affect the final results.

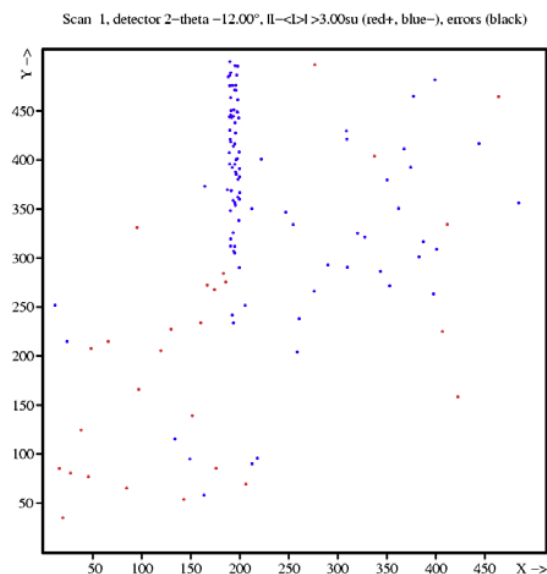


Figure 7.32 - A Spatial Distribution plot showing a problem area

7.3.5 Exit

1. Click on Exit AXScale to close the scaling GUI.



Figure 7.33 - Exit AXScale

NOTE: Scale is one of the few GUIs where it is necessary to exit using a special Exit button. This avoids an error message indicating that SADABS has exited improperly.

8. Examine Data

In this step, the space group for the sample is determined and optional simulated precession photographs are calculated to further evaluate the overall quality of the data. APEX2 provides two tools for this: XPREP for space group determination and Precession Images for looking at undistorted slices of reciprocal space. This is the final step before beginning the structure solution and refinement process.

8.1 XPREP

1. Click on XPREP (Space Group Determination) under Examine in the task bar.
2. Check that the two files in the pop-up window are correct (see Figure 8.1) and click OK.

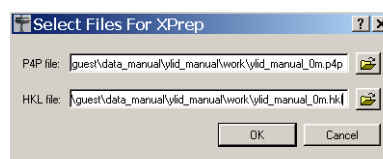


Figure 8.1 - Select files for XPREP input

NOTE: In this example, the scaling process has created two files: `ylid_manual_0m.p4p` containing the final unit cell parameters from integration and `ylid_manual_0m.hkl` containing the corrected intensities. Typically, these are the files to use for determining space groups, but you can browse to choose other files.

8.2 Space Group Determination

- XPREP evaluates the data and looks at the mean intensities and the mean int/sigma. Since these are large for all groups except P, XPREP suggests that the lattice is P (see Figure 8.2). Press Enter to accept.

```

XPREP Ver 6.14 W95/98/NT/2000/ME Copyright Bruker Nonius 2...
+-----+
+ XPREP - Reciprocal space exploration - Version 6.14 - W95/98/NT/2000/ME +
+ COPYRIGHT(c) 2003 Bruker Nonius All Rights Reserved +
+-----+

Screen size: 1280 x 1024
Window size: 640 x 923
Font size: 8 x 16 ( 125 x 178 )
Number of colors: 256

16154 Reflections read from file ylid_manual_Om.hkl
Mean (I/sigma) = 12.04

Lattice exceptions: P A B C I F Obv Rev All
N (total) = 0 8071 8097 8092 8088 12130 10773 10764 16154
N (int>3sigma) = 0 6377 6375 6480 6387 9616 8529 8517 12777
Mean intensity = 0.0 205.0 196.9 192.1 209.1 198.0 209.4 206.8 205.6
Mean int/sigma = 0.0 12.0 11.8 12.2 12.0 12.0 12.1 12.0 12.1

Lattice type [P, A, B, C, I, F, O(obv.), R(rev. rhomb. on hex. axes)]
Select option [P]:

```

Figure 8.2 - Lattice statistics

- XPREP determines the reduced cell based on the lattice entered above. Since the lattice was primitive and the magnitudes of the cell dimensions were proper ($a < b < c$), the original and reduced cells are the same (see Figure 8.3). Press Enter to search for a higher symmetry cell.

```

XPREP Ver 6.14 W95/98/NT/2000/ME Copyright Bruker Nonius 2...
Current dataset: ylid_manual_Om.hkl Wavelength: 0.71073 Chiral: ?
-----
Original cell: 5.957 9.027 18.366 90.00 90.00 90.00 Vol 987.7
Eads: 0.001 0.001 0.002 0.00 0.00 0.00 Lattice: P
-----
Current cell: 5.957 9.027 18.366 90.00 90.00 90.00 Vol 987.7
Matrix: 1.0000 0.0000 0.0000 0.0000 1.0000 0.0000 0.0000 0.0000 1.0000

[D] Read, modify or merge DATASETS [C] Define unit-cell CONTENTS
[P] Contour PATTERSON sections [F] Set up shelx1 FILES
[H] Search for HIGHER metric symmetry [R] RECIPOCAL space displays
[S] Determine or input SPACE GROUP [U] UNIT-CELL transformations
[A] Absorption, powder, SIR, SAD, MAD etc. [T] Change TOLERANCES
[M] Test for MEROHEDRAL TWINNING [O] Self-rotation function
[L] Reset LATTICE type of original cell [Q] QUIT program

Select option [H]:

```

Figure 8.3 - Reduced cell

For the YLID, no higher symmetry cell is found. The program has determined that the YLID crystal has an orthorhombic primitive lattice (see Figure 8.4). Press Enter to accept.

```

Determination of reduced (Niggli) cell
Transformation from original cell (HKL F-matrix):
1.0000 0.0000 0.0000 0.0000 1.0000 0.0000 0.0000 0.0000 1.0000
Unitcell: 5.957 9.027 18.366 90.00 90.00 90.00
Niggli form: a.a = 35.49 b.b = 81.49 c.c = 337.32
             b.c = 0.00 a.c = 0.00 a.b = 0.00

Search for higher METRIC symmetry
Identical indices and Friedel opposites combined before calculating R(sym)

Option A: FOM = 0.000 deg. ORTHORHOMBIC P-lattice R(sym) = 0.019 [ 4105]
Cell: 5.957 9.027 18.366 90.00 90.00 90.00 Volume: 987.68
Matrix: 1.0000 0.0000 0.0000 0.0000 1.0000 0.0000 0.0000 0.0000 1.0000
Option B retains original cell
Select option [A]:

```

Figure 8.4 - Higher symmetry cells

3. The next logical step is to determine the space group. XPREP suggests this (see Figure 8.5). Press Enter to determine the space group.

```

XPREP Ver 6.14 W95/98/NT/2000/ME Copyright Bruker Nonius 2...
Current dataset: ylid_manual_0m.hkl      Wavelength: 0.71073 Chiral: 2
-----
Original cell:  5.957  9.027 18.366  90.00  90.00  90.00  Vol: 987.7
             Eds:  0.001  0.001  0.002  0.00  0.00  0.00  Lattice: P
-----
Current cell:  5.957  9.027 18.366  90.00  90.00  90.00  Vol: 987.7
-----
Matrix: 1.0000  0.0000  0.0000  0.0000  1.0000  0.0000  0.0000  0.0000  1.0000
-----
Crystal system: Orthorhombic      Lattice: P
-----

[D] Read, modify or merge DATASETS      [C] Define unit-cell CONTENTS
[P] Contour PATTERSON sections          [F] Set up shelxtl FILES
[H] Search for HIGHER metric symmetry   [R] RECIPROCAL space displays
[S] Determine or input SPACE GROUP      [U] UNIT-CELL transformations
[A] Absorption, powder, SIR, SAD, MAD etc. [T] Change TOLERANCES
[M] Test for MEROHEDRAL TWINNING       [O] Self-rotation function
[L] Reset LATTICE type of original cell  [Q] QUIT program

Select option [S]:

```

Figure 8.5 - Determine the space group

4. If the space group is known or if the compound is known to be chiral, enter that information (see Figure 8.6). Generally, it is sufficient to press Enter to start the space group determination.

```

[S] Determine SPACE GROUP
[C] Must be CHIRAL (sample is optically active)
[N] NOT NECESSARILY chiral (eg. may be racemate)
[I] INPUT known space group
[E] EXIT to main menu or [Q] QUIT program

Select option [S]:

```

Figure 8.6 - Space Group options

5. XPREP has chosen the crystal system [O] (see Figure 8.7). Press Enter to accept.

```

[A] Triclinic, [M] Monoclinic, [O] Orthorhombic, [T] Tetragonal,
[H] Trigonal/Hexagonal, [C] Cubic or [E] EXIT

Select option [O]:

```

Figure 8.7 - Choose the crystal system

6. XPREP has chosen the crystal lattice [P] (see Figure 8.8). Press Enter to accept.

```

Lattice exceptions: P  A  B  C  I  F  Obv  Rev  All
N (total) =          0 8071 8097 8092 8088 12130 10773 10764 16154
N (int>3sigma) =      0 6377 6375 6480 6387 9616 8529 8517 12777
Mean intensity =    0.0 205.0 196.9 192.1 209.1 198.0 209.4 206.8 205.8
Mean int/sigma =    0.0 12.0 11.8 12.2 12.0 12.0 12.1 12.0 12.1

Lattice type [P, A, B, C, I, F, O(obv.), R(rev. rhomb. on hex. axes)]

Select option [P]:

```

Figure 8.8 - Choose the lattice

7. XPREP evaluates the data and looks at the systematic absences for all possible glide planes and screw axes (see Figure 8.9). These are displayed across the middle of the figure. By examining the number of reflections with $I > 3\sigma(I)$, the mean intensities, and the mean int/σ , which should all be very small for a systematic absence, XPREP derives a suggested space group, $P2(1)2(1)2(1)$. Press Enter to accept.

8.3 Reflection Statistics.

```

Mean |E*E-1| = 0.694 [expected .968 centrosym and .736 non-centrosym]

Systematic absence exceptions:

      b--  c--  n--  21--  -c-  -a-  -h-  -21-  --a  --b  --h  --21
N       761  768  759   18  437  434  443   28  308  314  306   39
N I>3s   572  557  549    0  319  325  280    0  239  253  216    2
<I>      225.1 238.3 274.2  0.3 457.1 405.7 201.4  0.6 277.4 337.8 291.0  0.8
<I/s>    12.3  11.8  12.1  0.3  14.2  14.0  10.9  0.5  13.8  14.9  13.3  0.6

Identical indices and Friedel opposites combined before calculating R(sym)

Option Space Group No. Type Axes CSD R(sym) N(eq) Syst. Abs. CFOM
[A] P2(1)2(1)2(1) # 19 chiral 1 5917 0.019 4105 0.6 / 10.9 1.14

Select option [A]:

```

Figure 8.9 - Systematic absences and a suggested space group

1. XPREP returns to the general menu seen previously (see Figure 8.5). This time D is chosen to evaluate the data set. Press Enter to accept.
2. There are multiple choices for data manipulation (see Figure 8.10). “S” chooses a display of statistics.

```

XPREP Ver 6.14 W95/98/NT/2000/ME Copyright Bruker Nonius 2...
Index # Data Filename or Source of Data
1 16154 ylid_manual_0m.hkl <- current dataset

[N] Sort-MERGE current data (no scaling) [C] Change CURRENT dataset
[L] LEAST-SQUARES scale and merge datasets [W] WRITE dataset to file
[I] INCLUDE Rfree flags from another file [R] READ in another dataset
[S] Display intensity STATISTICS [D] DELETE stored dataset
[F] FACE-indexed absorption corrections [P] PSI-scan absorption corr.
[T] Copy file, TRANSFORM hkl and cosines [A] MAD, SAS, SIR or SIRAS
[H] Apply HIGH/low resolution cutoffs [N] NORMALIZE/scale sigmas
[G] Generate simulated powder diagrams [E] EXIT to main menu
[Q] QUIT program

Select option [S]:

```

Figure 8.10 - The data manipulation menu

3. The data can be merged in several ways. Choose the “Merge ALL equivalents including Friedel opposites” option [A] (see Figure 8.11). Press Enter to accept.

NOTE: This merge will not average the reflections in the final data file. It is only for the calculation of statistics.

```

XPREP Ver 6.14 W95/98/NT/2000/ME Copyright Bruker Nonius 2...
Current dataset: ylid_manual_0m.hkl

[N] Do NOT merge reflections
[I] Merge IDENTICAL indices only
[S] Merge SYMMETRY equivalents (but not Friedel opposites)
[A] Merge ALL equivalents (including Friedel opposites)
[E] EXIT to merge datasets menu
[Q] QUIT program

Select option [A]:

```

Figure 8.11 - The merge data menu

4. A table of statistics appears (see Figure 8.12). Examine the data presented. Is the completion near 100%? Is the redundancy good? Are Rint and Rsigma small and increasing smoothly from top to bottom? In particular, look at the last two lines which compare all of the data with the high-resolution shell and the complete data set. The completion should be near 100% for both the high-resolution shell and the complete data set. The redundancy and Rint should be similar for the two. The Mean Intensity and the Mean I/sigma(I) will usually be quite different. The Mean I/sigma(I) for the high-resolution data should be greater than 3.0.

For this data set, when integrated to 0.67Å resolution, the difference between the Rint for the high-resolution data and the complete data set is slightly larger than expected. Press Enter to continue.

XPREF Ver 6.14 W95/98/NT/2000/ME Copyright Bruker Nonius 2...

Resolution	#Data	#Theory	%Complete	Redundancy	Mean I	Mean I/s	Rint	Rsigma
Inf - 1.95	105	105	100.0	12.07	1140.8	71.33	0.0295	0.0118
1.95 - 1.50	105	105	100.0	10.94	495.4	66.41	0.0266	0.0151
1.50 - 1.30	108	108	100.0	11.38	276.9	65.26	0.0329	0.0145
1.30 - 1.15	126	126	100.0	10.11	211.2	56.76	0.0327	0.0157
1.15 - 1.05	134	134	100.0	9.61	119.8	46.09	0.0361	0.0174
1.05 - 0.95	176	176	100.0	8.94	92.5	40.47	0.0467	0.0221
0.95 - 0.90	125	125	100.0	8.56	63.1	32.54	0.0580	0.0262
0.90 - 0.85	148	148	100.0	8.14	43.2	24.60	0.0743	0.0352
0.85 - 0.80	199	199	100.0	7.23	37.0	20.31	0.0822	0.0441
0.80 - 0.75	236	236	100.0	6.67	27.7	14.66	0.1113	0.0638
0.75 - 0.70	327	327	100.0	5.92	18.4	8.81	0.1506	0.1083
0.70 - 0.67	248	256	96.9	4.50	10.8	4.51	0.2104	0.2361
0.80 - 0.67	811	819	99.0	5.69	18.7	9.20	0.1408	0.1116
Inf - 0.67	2037	2045	99.6	7.90	146.0	30.03	0.0359	0.0206

Merged [A], lowest resolution = 18.37 Angstroms, 650 outliers downweighted

Enter <CR> to continue

Figure 8.12 - Intensity statistics

8.4 Applying High Resolution Cutoff

NOTE: For the YLID crystal, if the resolution limit for integration was changed to 0.75 as suggested on Page 7-3, then there will be no need to apply a High Resolution Cutoff at this point.

1. Since the difference between the Rint for the high-resolution data and the complete data set is slightly larger than expected, it is reasonable to apply a high-resolution cutoff using the H option in the data manipulation menu. (see Figure 8.13). Type H and press Enter.

```
[M] Sort-MERGE current data (no scaling)      [C] Change CURRENT dataset
[L] LEAST-SQUARES scale and merge datasets    [W] WRITE dataset to file
[I] INCLUDE Pfree flags from another file     [R] READ in another dataset
[S] Display intensity STATISTICS              [D] DELETE stored dataset
[F] FACE-indexed absorption corrections       [P] PSI-scan absorption corr.
[T] Copy file, TRANSFORM hkl and cosines     [A] MAD, SAS, SIR or SIRAS
[H] Apply HIGH/low resolution cutoffs         [N] NORMALIZE/scale sigmas
[G] Generate simulated powder diagrams        [E] EXIT to main menu
[Q] QUIT program

Select option [E]: H
```

Figure 8.13 - Requesting a high resolution cutoff

2. Enter a high resolution limit of 0.75 (see Figure 8.14) and press Enter to accept the low resolution cutoff of infinity.

```
Select option [E]: h

High resolution limit in Angstroms [1.0]: 0.75

Low resolution limit [inf]:
```

Figure 8.14 - The resolution cutoff menu

3. Applying the cutoff removes approximately 3000 reflections from the data set (see Figure 8.15). The next step is to see if the cutoff has improved the statistics. Type S and press Enter.

```

XPREP Ver 6.14 W95/98/NT/2000/ME Copyright Bruker Nonius 2...
Index # Data Filename or Source of Data
1 16154 ylid_manual_0m.hkl
2 13067 ylid_manual_0m.hkl <- current dataset

[N] Sort-MERGE current data (no scaling) [C] Change CURRENT dataset
[L] LEAST-SQUARES scale and merge datasets [W] WRITE dataset to file
[I] INCLUDE Rfree flags from another file [R] READ in another dataset
[S] Display intensity STATISTICS [D] DELETE stored dataset
[F] FACE-indexed absorption corrections [P] PSI-scan absorption corr.
[T] Copy file, TRANSFORM hkl and cosines [A] MAD, SAS, SIR or SIRAS
[H] Apply HIGH/low resolution cutoffs [N] NORMALIZE/scale sigmas
[G] Generate simulated powder diagrams [E] EXIT to main menu
[Q] QUIT program

Select option [E]:

```

Figure 8.15 - Requesting statistics after applying a high resolution cutoff

4. As in step 3 of Section 8.3, enter [A] to merge all equivalents.

5. After the cutoff, all of the statistics look better (see Figure 8.16). Press Enter to continue.

Resolution	#Data	#Theory	%Complete	Redundancy	Mean I	Mean 1/s	Rint	Rsigma
Inf - 2.25	74	74	100.0	12.59	1369.5	73.60	0.0292	0.0116
2.25 - 1.70	82	82	100.0	11.18	584.2	66.45	0.0284	0.0145
1.70 - 1.45	82	82	100.0	10.84	414.1	67.42	0.0285	0.0142
1.45 - 1.30	80	80	100.0	11.34	231.3	62.58	0.0342	0.0152
1.30 - 1.15	126	126	100.0	10.11	211.2	56.76	0.0327	0.0157
1.15 - 1.05	134	134	100.0	9.61	119.8	46.09	0.0361	0.0174
1.05 - 0.95	176	176	100.0	8.94	92.5	40.47	0.0467	0.0221
0.95 - 0.90	125	125	100.0	8.56	63.1	32.54	0.0580	0.0262
0.90 - 0.85	148	148	100.0	8.14	43.2	24.60	0.0743	0.0352
0.85 - 0.80	199	199	100.0	7.23	37.0	20.31	0.0822	0.0441
0.80 - 0.75	236	236	100.0	6.67	27.7	14.66	0.1113	0.0638
0.75 - 0.70	435	435	100.0	6.92	31.9	17.25	0.0949	0.0534
Inf - 0.75	1462	1462	100.0	8.94	197.5	39.10	0.0339	0.0168
Merged [A], lowest resolution = 18.37 Angstroms, 610 outliers downweighted								
Enter <CR> to continue								

Figure 8.16 - Statistics after high resolution cutoff

6. This response will return to the data manipulation menu (see Figure 8.10, Figure 8.13 or Figure 8.15). The default answer should be E for Exit. Press Enter to accept this default and return to the general menu.

8.5 Preparing an Output File

1. In the general menu, chose C to define the unit cell contents.
2. A window will open displaying the current formula, Z, the density, and the atomic volume (see Figure 8.17). In this example, the formula is incorrect and Z has been set to six to try to achieve a reasonable density and atomic volume. Since this formula is incorrect, it must be modified now. The correct chemical formula for the YLID crystal is $C_{11}H_{10}O_2S$.

2.1 At Select Option, do not accept the default answer of E. Type in F to enter a new formula.

2.2 In response to the question "Enter Formula," type the correct formula C11 H10 O2 S1 and press Enter.

2.3 Check that the information is correct. Check that Z seems reasonable for the space group, that the density is as expected (1.1 to 1.4 for organic molecules, higher for inorganic compounds), and that the atomic volume is around 17 or 18. Significant variation from the expected values may indicate an incorrect molecular formula or missing counter ions or solvates. The values at the bottom of the window look fine for the YLID. Press Enter to accept.



Figure 8.17 - Defining and checking the unit cell contents

3. The next default action for XPREP is to write out the files necessary for the structure solution process (see Figure 8.18). Press Enter to accept.

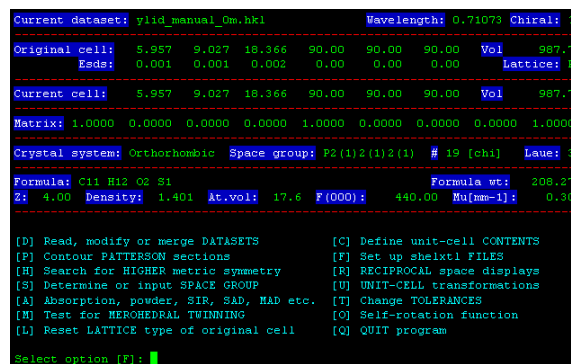


Figure 8.18 - Requesting output files

4. The program asks for an output file name. Since this data has been cut off at a resolution of 0.75Å, it makes sense to change the output file name to ylid_res75 to reflect the cutoff (see Figure 8.19). Press Enter to accept.

```
Current dataset: ylid_manual_0m.hkl      Wavelength: 0.71073 Chiral: ?
-----
Original cell:  5.957  9.027 18.366  90.00  90.00  90.00  Vol: 987.7
Eads: 0.901  0.001  0.002  0.00  0.00  0.00  Lattice: P
-----
Current cell:  5.957  9.027 18.366  90.00  90.00  90.00  Vol: 987.7
-----
Matrix: 1.0000  0.0000  0.0000  0.0000  1.0000  0.0000  0.0000  0.0000  1.0000
-----
Crystal system: Orthorhombic Space group: P2(1)2(1)2(1) # 19 [ch] Laue: 3
-----
Formula: C11 H12 O2 S1      Formula wt: 208.27
Z: 4.00 Density: 1.401 At.vol: 17.6 F(000): 440.00 Mu[mm-1]: 0.30
-----

[D] Read, modify or merge DATASETS      [C] Define unit-cell CONTENTS
[P] Contour PATTERSON sections          [F] Set up shelxtl FILES
[H] Search for HIGHER metric symmetry  [R] RECIPROCAL space displays
[S] Determine or input SPACE GROUP     [U] UNIT-CELL transformations
[A] Absorption, powder, SIR, SAD, MAD etc. [T] Change TOLERANCES
[M] Test for MEROHEDRAL TWINNING      [O] Self-rotation function
[L] Reset LATTICE type of original cell [Q] QUIT program

Select option [F]:
Output file name (without extension) [ylid_manual_0m]: ylid_res75
```

Figure 8.19 - Changing the file name

5. After entering the file name (or hitting Enter to accept the default file name) an input file for the structure solution module is created and displayed on the screen, and the program asks, "Do you wish to (over)write the intensity data file ylid_res75.hkl?" Since the file name has been changed, this question **must** be answered with a y (see Figure 8.20).

```
Select option [F]:
Output file name (without extension) [ylid_manual_0m]: ylid_res75

File ylid_res75.ins set up as follows:

TITL ylid_res75 in P2(1)2(1)2(1)
CELL 0.71073  5.9573  9.0270 18.3664  90.000  90.000  90.000
ZERR  4.00  0.0005  0.0008  0.0019  0.000  0.000  0.000
LATT  -1
SYMM 0.5-X, -Y, 0.5+Z
SYMM -X, 0.5+Y, 0.5-Z
SYMM 0.5+X, 0.5-Y, -Z
SFAC C H O S
UNIT 44 48 8 4
TEMP 0
TREF
HKL 4
END

Do you wish to (over)write the intensity data file ylid_res75.hkl ? [N]: y
```

Figure 8.20 - The input file for structure solution and a final question

6. Exit XPREP (see Figure 8.21). Press Enter to exit the program.

```
[D] Read, modify or merge DATASETS      [C] Define unit-cell CONTENTS
[P] Contour PATTERSON sections          [F] Set up shelxtl FILES
[H] Search for HIGHER metric symmetry  [R] RECIPROCAL space displays
[S] Determine or input SPACE GROUP     [U] UNIT-CELL transformations
[A] Absorption, powder, SIR, SAD, MAD etc. [T] Change TOLERANCES
[M] Test for MEROHEDRAL TWINNING      [O] Self-rotation function
[L] Reset LATTICE type of original cell [Q] QUIT program

Select option [Q]:
```

Figure 8.21 - Exit XPREP from the general menu

NOTE: There are many other features in XPREP that can be accessed from the general menu; reciprocal space plots, simulated powder patterns and a test for merohedral twinning are very useful tools.

8.6 Simulated Precession Images

1. Click on Precession Images under Examine in the task bar. Precession Images provide an undistorted view of layers of the reciprocal lattice. APEX2 generates simulated precession images by finding the appropriate pixels in a series of “.sfrm” images. You must specify the images to examine and the zones to calculate.
2. To open the File Selection window, left-click on the folder beside the file name. Choose the sets of images that you want to use for the calculation by clicking on the check boxes (see Figure 8.22).

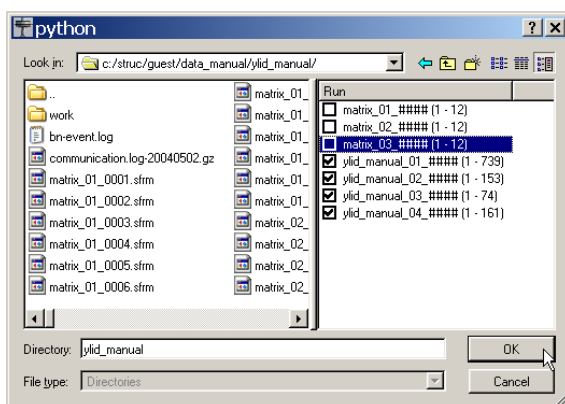


Figure 8.22 - File Selection window with matrix runs deselected

3. Check the default input value. Modify as needed and press Calculate.

Calculating more zones does not significantly increase the calculation time. In the example, 1kl, h1l and hk1 have been added to the defaults of 0kl, h0l and hk0. Fractional values (e.g., 0.5kl) are allowed. Reducing the resolution may speed the calculations slightly. The thickness defines the range of pixels above and below the requested range. For example, if the 0kl zone is requested with a thickness of 0.1, then the simulation is looking for all pixels that have $-0.1 < h < 0.1$, and any value (including fractional values) for k and l.

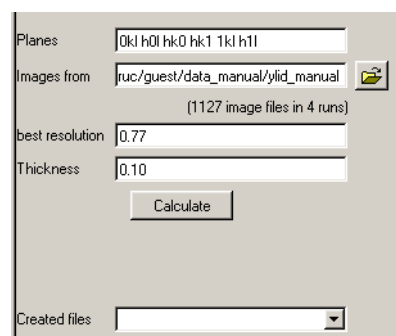


Figure 8.23 - Detail of the input section

4. A progress bar will appear (Figure 8.24) and after approximately 5–10 minutes (depending on the number of “.sfrm” files read) an image will appear on the screen. Display other planes by clicking on the calculated images to the bottom right of the work area or by browsing as usual with the View plug-in.

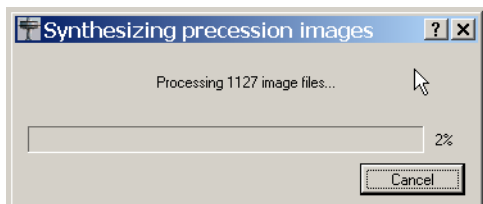


Figure 8.24 - Progress bar

5. Use the simulated patterns to check space group symmetry (see Figure 8.25, Figure 8.26 and Figure 8.27) and to find signs of twinning (see Figure 8.28).

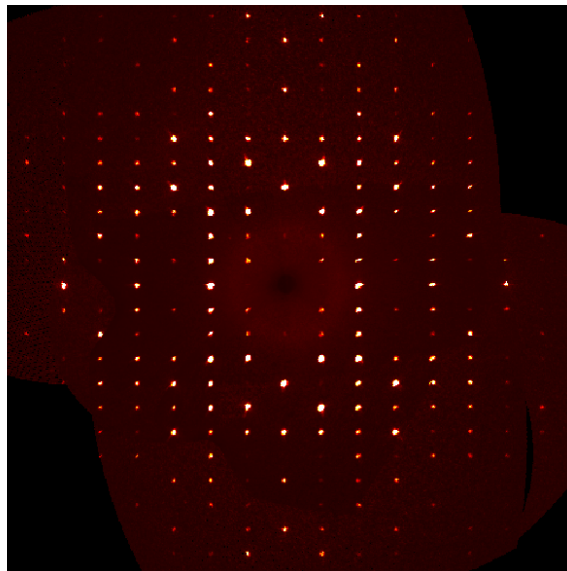


Figure 8.25 - The 0kl plane for the test YLID

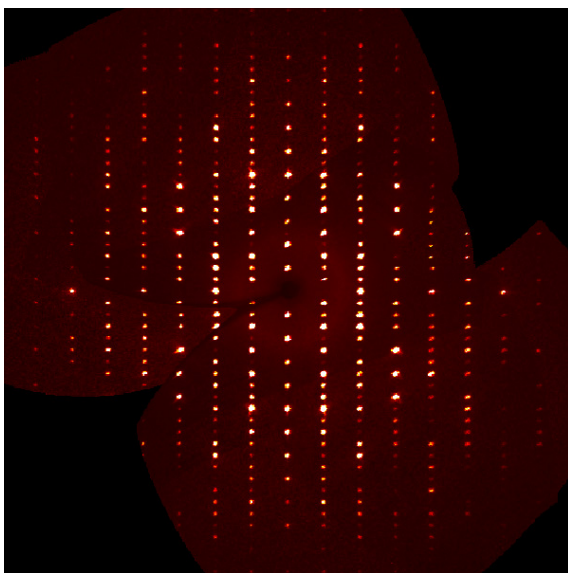


Figure 8.26 - The h0l plane for the test YLID

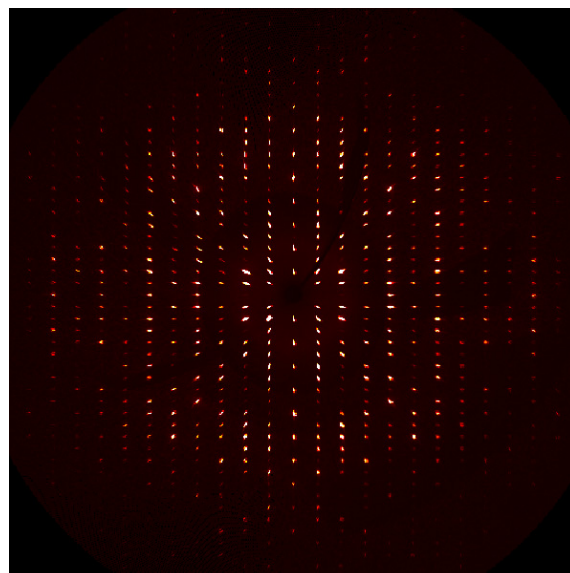


Figure 8.27 - The hk0 plane for the test YLID

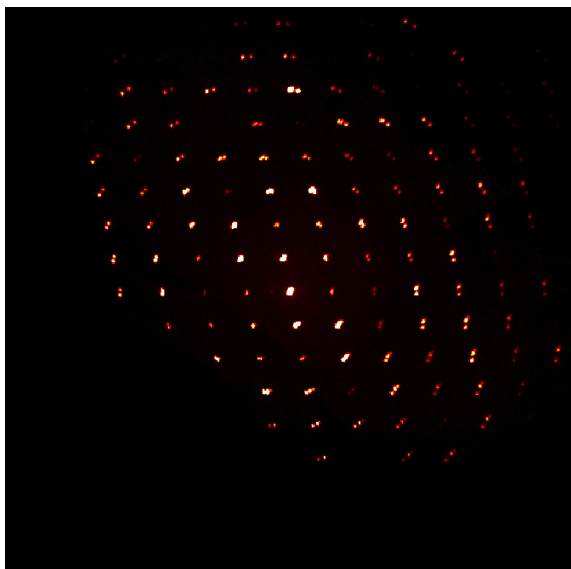


Figure 8.28 - A plane from a rotationally twinned crystal showing the two lattices

9. Structure Solution and Refinement

9.1 Overview

You are now ready to solve and refine the crystal structure. The various steps in solving and refining the structure are carried out within the APEX2 GUI. XPREP has prepared the input files for a standard direct methods run. There are two files: .ins and .hkl. In this example, the two files are ylid_res75.ins and ylid_75.hkl.

These files are all that is required to begin the structure solution and refinement process. The various steps of solving and refining the structure are carried out using the Solve and Refine functions of the APEX2 Suite.

9.2 Solve the Structure

The process of obtaining an initial model of the compound is started by clicking in the Task Bar on Solve Structure and then on Structure Solution.

1. This will open the Structure Solution GUI. Check that there is a reasonable formula in the formula window.

- 1.1 If you entered a correct formula in the display GUI, then you are ready to start a normal direct methods run.
- 1.2 If the formula is incorrect, change it in the formula window and press Return. The input file will update.

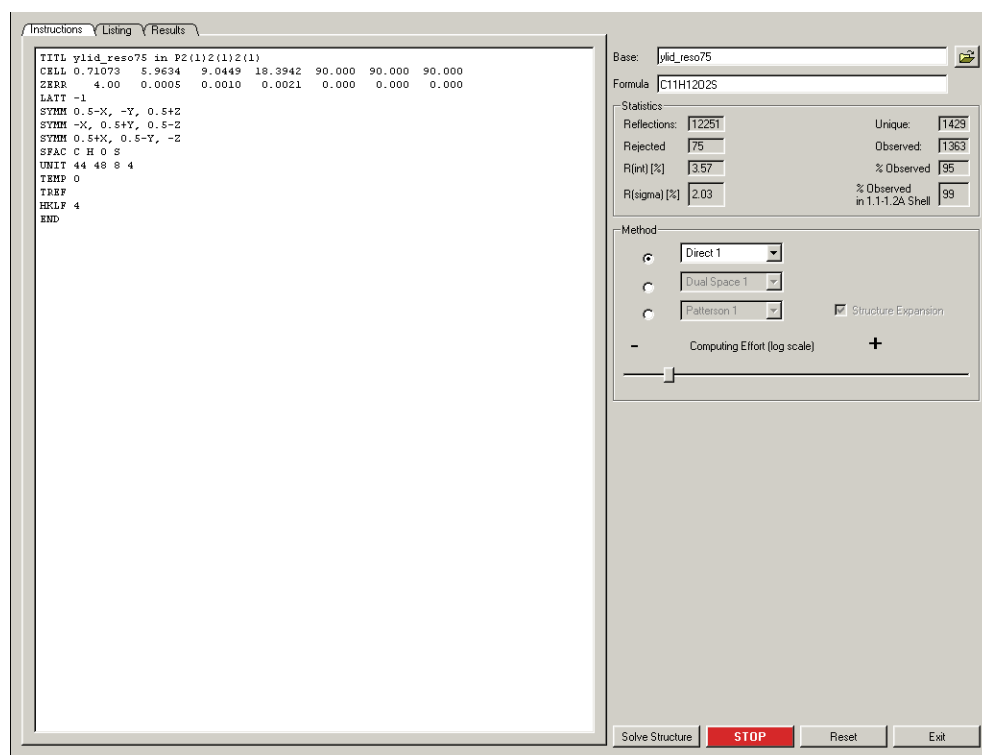


Figure 9.1 - Default Solve Structure view

- Click on Solve Structure to start the calculations and open an output window. This window can be used to monitor the progress of the direct methods calculations.

Structure Solution Log Window

```

172 Reflections and 3346 unique TPR for R(alpha)
465 Unique negative quartets found, 465 used for phase refinement

110 Unique NQR employed in phase annealing
128 Parallel refinements, highest memory = 3822 / 44055

Try Ralpha Nqual Sigma-1 M(abs) CFOM Seminvariants

1890781. 0.064 -0.770 0.946 1.154 0.064* +--+ +--+ +--+
Freq: 0 0 0 97 13 0 0 0 0 0 0 0 0 1 0 0 1 0 0 1 2 1 1 1 / 128

917713. 0.060 -0.799 0.946 1.151 0.060* +--+ +--+ +--+
Freq: 0 0 1 201 25 0 0 0 0 0 0 0 1 0 0 1 0 1 2 2 1 0 0 1 2 2 1 1 / 256

256. Phase sets refined - best is code 917713. with CFOM = 0.0597

Fourier and peaksearch
RE = 0.154 for 14 atoms and 467 E-values
Fourier and peaksearch
RE = 0.140 for 14 atoms and 467 E-values
Fourier and peaksearch

+++++
+ ylid_reso75 finished at 07:46:22 Total elapsed time: 0.9 secs +
+++++

```

Figure 9.2 - Structure Solution output

- Click on the arrow to the right of Direct 1 in the Method box (see Figure 9.3). This gives three choices for structure solution using direct methods.

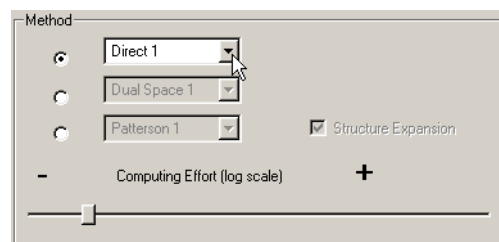


Figure 9.3 - Method box

In most cases the default values will give a good initial model, but there are several other options available for more difficult problems.

- Chose the solution method by clicking the appropriate radio button on the left in the Method box.
- Highlight and click on Direct 1 to set up a default run with a simple TREF instruction.
- Highlight and click on Direct 2 to set up an extended run with TREF 10000. By adding 10000 to the TREF command, more attempts are made to determine a solution. This will take more time, but if you see a good solution in the output window it is possible to stop at that point by clicking on the STOP button. Good solutions typically have a CFOM of 0.06 or less.
- Highlight and click on Direct 3 to set up an extreme run with two instructions, ESEL 1.0 and TREF 100000. By adding ESEL 1.0, more reflections are used in the solution process. Increasing the number of tries in the TREF command to 100000 runs (until ended by clicking on the STOP button) is particularly useful for acentric triclinic structures and for pseudo-symmetric structures. There is only one pre-defined option for the dual space calculations and also only one for the heavy atom Patterson methods.

At the end of the direct methods runs, the output display will look like Figure 9.2. Good figures of merit are near 1.0 for Sigma-1 and M(abs) and less than 0.06 for Ralpha and the CFOM. Structures can be solved with figures of merit that deviate from these numbers, but they may require more effort.

If the initial model looks reasonable, click OK in the output display and Exit in the Solutions plug-in. The next step is structure refinement using XShell.

9.3 XShell

9.3.1 Refine the Structure

The structure solution step produced statistics indicating that a solution had been found. The real proof, however, is in the initial model that is produced. XShell provides the tools to view and refine the model. A quick glance at the results of the direct methods run is often all that is needed to see that the results make chemical sense. Control of the refinement process is quite straightforward using XShell.

1. Start XShell, the molecular viewing and refinement plug-in, by clicking on Refine Structure in the Task Bar and then on the Structure Refinement icon.
2. This will open a window to select files for XShell. In usual operations, the file name will be pre-selected. If a different file is desired, select Browse.

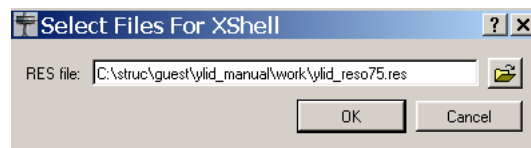
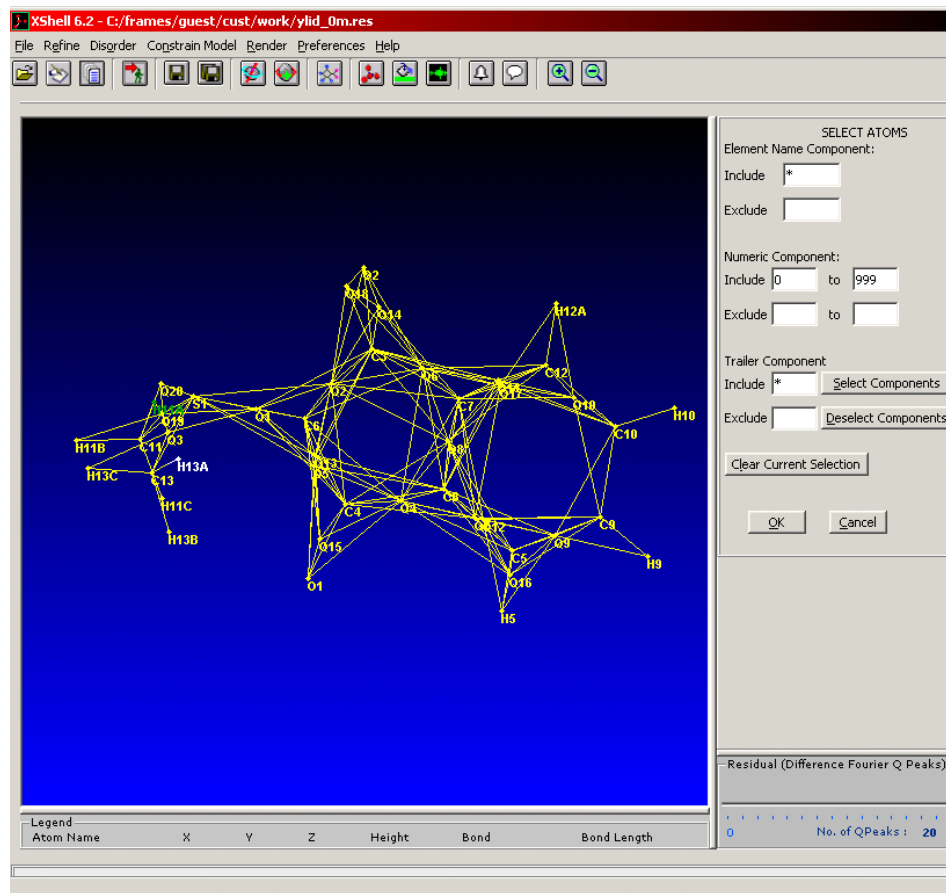


Figure 9.4 - The Select Files tool

- Click on OK to open a molecular display window.



4. Right-click in the background to open a pop-up window with options to change the image.

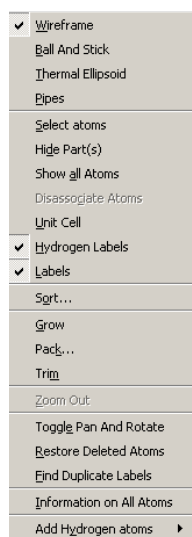


Figure 9.6 - XShell menu

5. Click on the Information On All Atoms option to open a window displaying a list of the peaks and their heights (see Figure 9.7). The drop in height between Q13 (the last real peak) and Q14 (the first noise peak) is typical of a correct solution. Click on OK to close this window.

Information On All Atoms

INFORMATION ON ALL ATOMS									
	LABEL	PEAK HEIGHT	TYPE	X	Y	Z	HYBRID	CHARGE	UEQ
1	S1	0.0000	S	0.8105	0.1820	0.2401	sp3	0	0.05
2	Q1	161.0300	Q	0.6925	0.10	0.1253	sp3	0	0.05
3	Q2	158.7400	Q	0.3333	0.3057	0.1756	sp3	0	0.05
4	Q3	155.9800	Q	0.4332	0.1961	0.1488	sp3	0	0.05
5	Q4	141.7900	Q	0.5055	0.3	0.685	sp3	0	0.05
6	Q5	141.6800	Q	0.8344	0.898	0.1307	sp3	0	0.05
7	Q6	140.8200	Q	0.6372	0.1262	0.1726	sp3	0	0.05
8	Q7	140.6700	Q	0.3537	0.1161	0.834	sp3	0	0.05
9	Q8	127.0800	Q	0.1821	0.1416	0.360	sp3	0	0.05
10	Q9	109.9200	Q	0.8324	0.3761	0.2281	sp3	0	0.05
11	Q10	109.4100	Q	0.1499	0.486	0.244	sp3	0	0.05
12	Q11	104.8200	Q	0.2908	0.615	0.380	sp3	0	0.05
13	Q12	98.8900	Q	0.4789	0.856	0.93	sp3	0	0.05
14	Q13	85.8200	Q	0.6561	0.1836	0.3232	sp3	0	0.05
15	Q14	66.1600	Q	0.5674	0.1814	0.2395	sp3	0	0.05
16	Q15	64.7300	Q	0.8062	0.4621	0.2409	sp3	0	0.05
17	Q16	54.7300	Q	0.9493	0.1856	0.1563	sp3	0	0.05
18	Q17	40.2200	Q	0.52	0.989	0.237	sp3	0	0.05

OK Print

Figure 9.7 - Atom information screen

At the bottom right of the main window is a slide bar which is used for deselecting peaks. The arrow can be moved by left-clicking and dragging the arrow or by left-clicking on either side of the arrow. Left-clicking to the left of the arrow (i.e., where the mouse cursor is pointed in Figure 9.8) will remove peaks.

- Slide the pointer down while watching the molecular display. Change the number of Q peaks to 15.

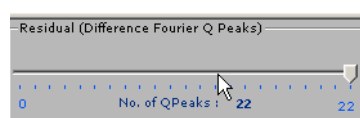


Figure 9.8 - The Q peak slider

At 15 the molecule is much cleaner, but there are still peaks that do not make sense.

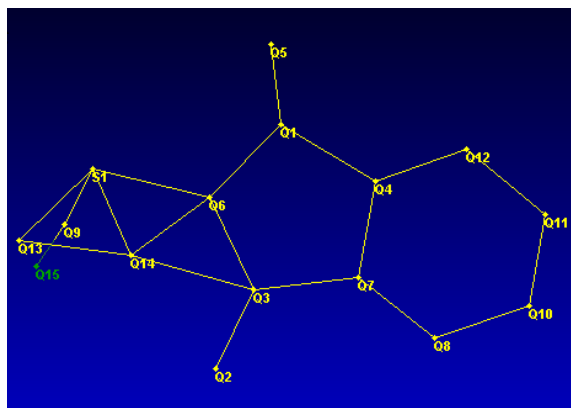


Figure 9.9 - Image after the number of Q peaks was changed to 15

Click left on the slider until you find a reasonable model.

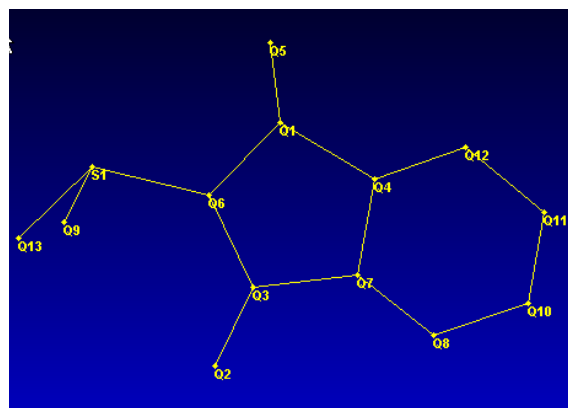


Figure 9.10 - Image with all noise peaks removed

7. Put the cursor over the bonds to check distance. The distance will be displayed at the bottom right.

9.3.2 Label the Atoms

1. Label the atoms and set the atom types.
Left-click on atoms to select them. Left-click on the peaks for the two oxygen atoms (Q2 and Q5 in this example).

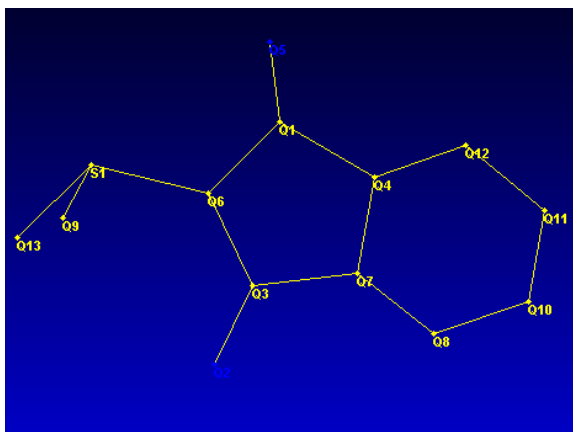


Figure 9.11 - View of model with the probable oxygen peaks selected

2. If it is difficult to see the color and labels, change the color scheme by left-clicking on Preferences and then right-clicking on Background Color. Choose colors and click Apply. Click Cancel to exit the background color mode. Note that the selected peaks are blue in Figure 9.13.

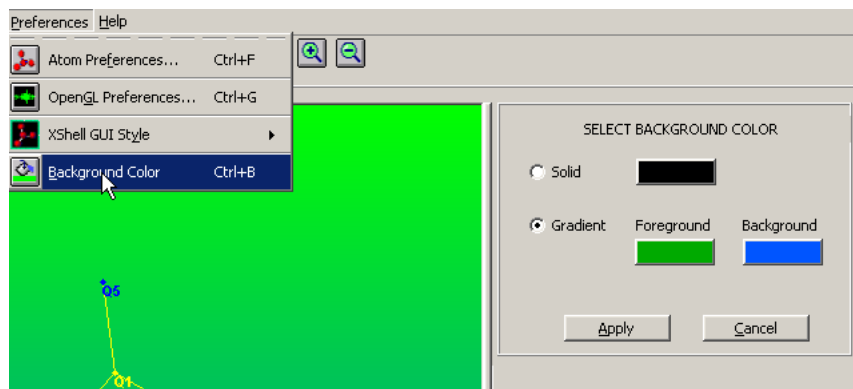


Figure 9.12 - Selecting the background color

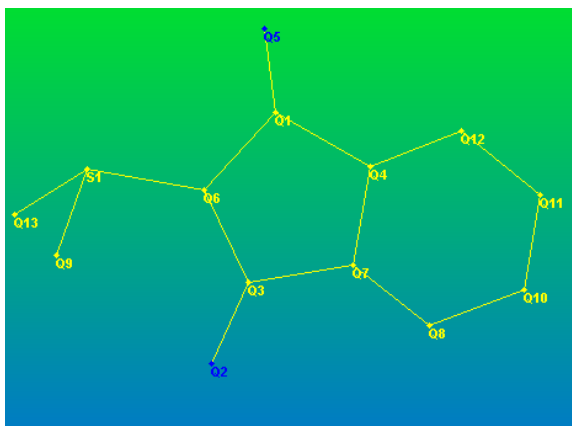


Figure 9.13 - New background color

3. Right-click to view a pop-up menu and select Labelling.

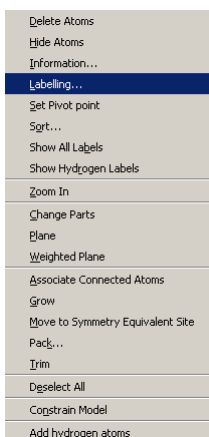


Figure 9.14 - Select Labelling

The Atom Labelling box will open.

Figure 9.15 - Atom Labelling box

4. The two selected atoms need to be changed to oxygen atoms. Do this one of two ways:
 - Click on the Element field and type in the element symbol (case does not matter).
 - Click on the El button to the right of the Element field. This will open a periodic table. Left-click on the appropriate element symbol to select it (the periodic table will automatically close).

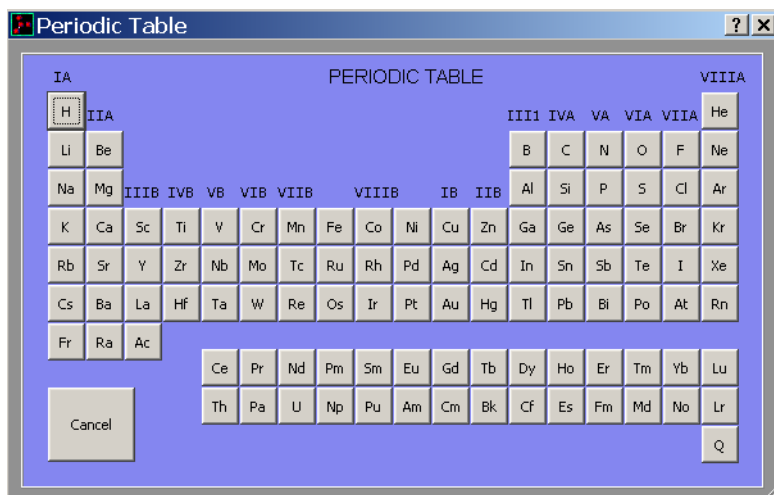


Figure 9.16 - The periodic table for selecting atom types

5. Once the element type is set correctly, click on Relabel to number the peaks sequentially in the order they were selected, starting with the number in the First Sequence # field.

6. Seeing the color of atoms can be difficult in the Wireframe view used so far. Right-click in the background with no atoms selected to get a slightly different action menu. In this menu, highlight and left-click on Pipes. This makes the bonds thicker and makes it obvious that there is an incorrectly labeled atom. This will be corrected when the carbon atoms are labeled.

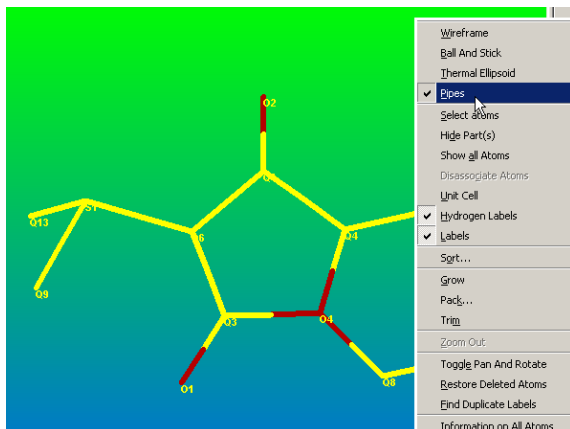


Figure 9.17 - Choose the pipes view

7. Left-click on the remaining peaks in the order that you want them labeled.

NOTE: If working on YLID test data, look at Figure 9.18 and click on the peaks to give the same order as used here. Right-click and choose Labelling. Select Carbon and apply the labels.

8. In the Labelling tool, change the element type to C (carbon). The starting atom number will change to 1—the first available number for carbon atoms.
9. Click Relabel.
10. Click Cancel to close the window.

9.3.3 Refine the Molecule

The labeled YLID molecule is now ready for refinement.

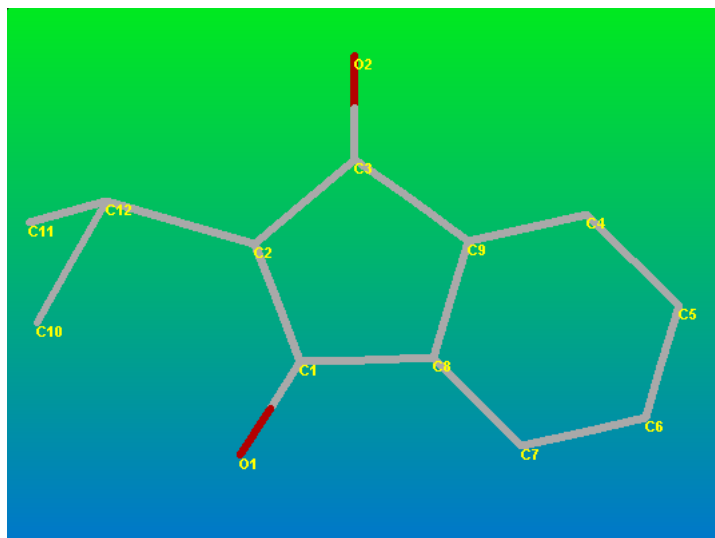


Figure 9.18 - Correctly labeled model

1. Left-click on the Refine icon in the tool icon bar (the cursor is pointing to it in Figure 9.19) or Type Control+R or go to Refine in the Windows tool bar.



Figure 9.19 - The Refine icon

This will open the Refine Box. Click on Refine to launch XL (the least-squares refinement program) using the default parameters as given.

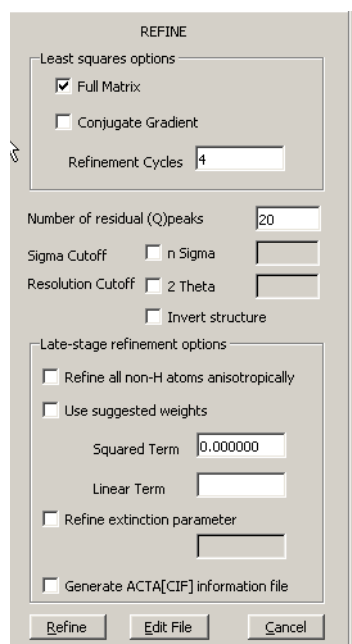


Figure 9.20 - The Refine menu

After a few seconds, a Refine window will open with output results for the calculation.

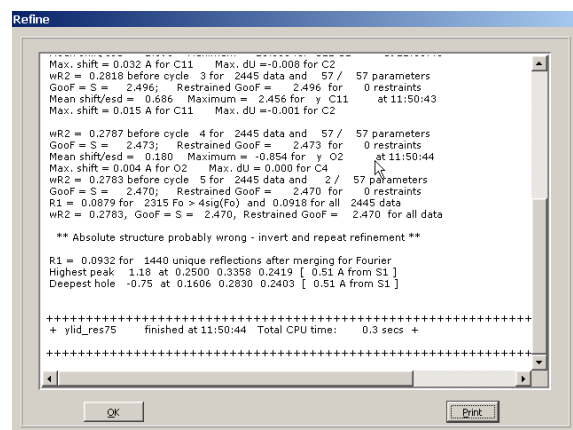


Figure 9.21 - Isotropic refinement output

In the output displayed above, note that the R1 value is 0.08. This is typical for a preliminary isotropic refinement for an organic molecule with data to a resolution of 0.75 and no hydrogen atoms included. The refinement program also indicates that the model needs to be inverted to get the correct absolute structure.

NOTE: This indication of the absolute configuration is usually reliable, but all assignments of absolute structure should be confirmed later in the refinement process by including TWIN and BASF cards in the instruction input.

- Click on OK to return to XShell. The molecule display will refresh with the results of the least-squares calculations.

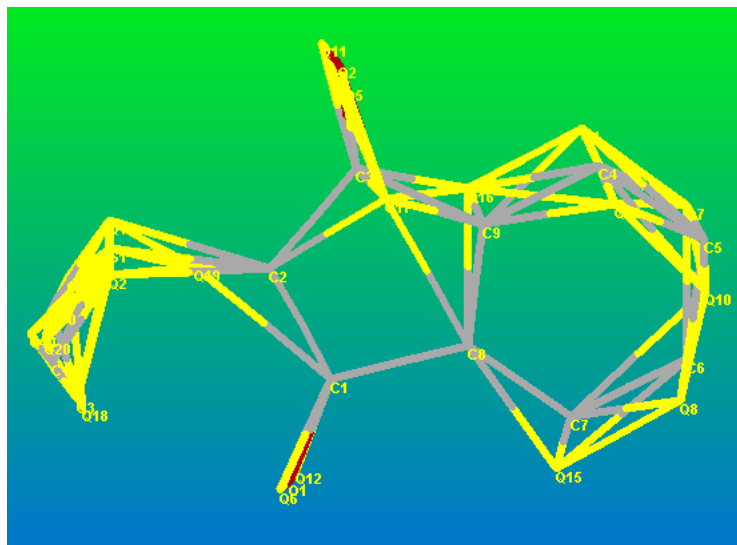


Figure 9.22 - The model after isotropic refinement

The peaks in the diagram represent difference in the electron density between the refinement model and the experimental electron density as defined by the measured data. Many of these difference peaks are near the sulfur and oxygen atoms. Refinement of all atoms anisotropically should improve the model.

- In the refinement box, click on Invert Structure and Refine All Non-H Atoms Anisotropically as in Figure 9.23.

Figure 9.23 - Inverting and choosing anisotropic refinement

- Click on the Refine button to launch the least-squares refinement program. The output window will open and a summary of the results of individual cycles of refinement will appear.

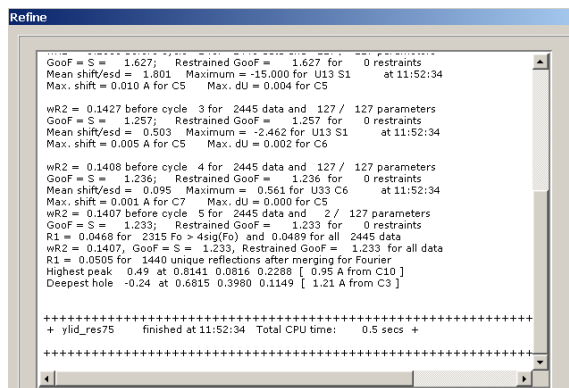


Figure 9.24 - Least-squares refinement summary

The value for R1, 0.047, is typical for an initial anisotropic refinement with no hydrogen atoms included. Since the mean shift/esd is greater than 0.1, the model is still changing. Since the highest peak in the difference map has a height of 0.49 and is 0.5 Å from C10, there are certainly some hydrogen atoms to be included.

Examination of the difference peaks in the resulting difference electron density map shows that the top difference peaks all correspond to hydrogen atoms.

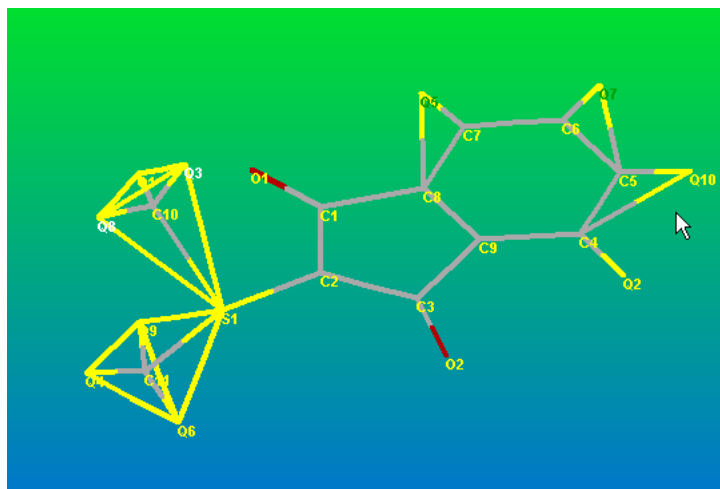


Figure 9.25 - Difference peaks correspond to expected hydrogen atom positions

9.3.4 Look at Atomic Displacement Parameters (Thermal Ellipsoids)

It is generally good practice to examine the displacement parameters at this point in structure determination.

1. Remove difference peaks with the slider tool.

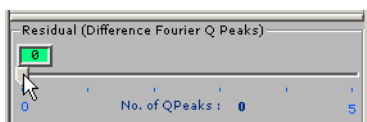


Figure 9.26 - Removing all Q peaks with the slider bar

2. Right-click on the background and select Thermal Ellipsoids.

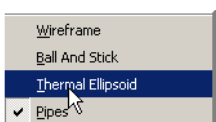


Figure 9.27 - Selecting Thermal Ellipsoids

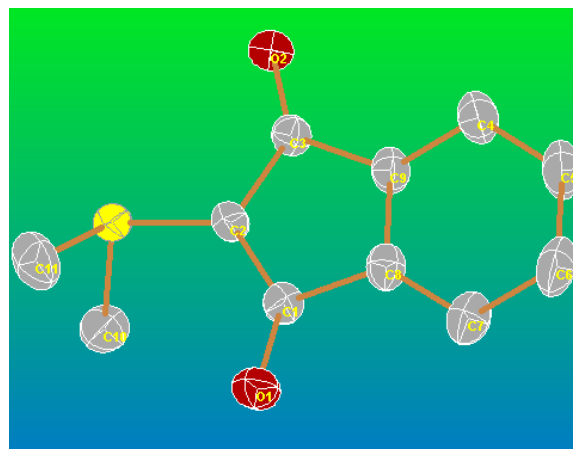


Figure 9.28 - Thermal Ellipsoid plot

Alternatively, a list of Ueq's (equivalent isotropic displacement parameters) can be generated by requesting information on all of the atoms. Examining these values for outliers is good practice.

3. Right-click on the background and click on Information on All Atoms.

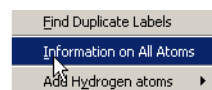


Figure 9.29 - Selecting Information on All Atoms

This produces the table in Figure 9.30.

Information On All Atoms

INFORMATION ON ALL ATOMS									
	LABEL	PEAK HEIGHT	TYPE	X	Y	Z	HYBRID	CHARGE	UEQ
1	S1	0.0000	S	0.8097	0.1815	0.2405	sp3	0	0.0353467
2	O1	0.0000	O	0.3345	0.3026	0.1765	sp3	0	0.04722
3	O2	0.0000	O	0.8432	0.890	0.1287	sp3	0	0.0488033
4	C8	0.0000	C	0.3591	0.1141	0.827	sp3	0	0.03482
5	C6	0.0000	C	0.1496	0.502	0.228	sp3	0	0.0549167
6	C3	0.0000	C	0.6896	0.8	0.1251	sp3	0	0.03449
7	C1	0.0000	C	0.4329	0.1949	0.1500	sp3	0	0.0335967
8	C9	0.0000	C	0.5093	0.24	0.679	sp3	0	0.0371967
9	C2	0.0000	C	0.6354	0.1245	0.1726	sp3	0	0.0332767
10	C7	0.0000	C	0.1756	0.1410	0.377	sp3	0	0.0462133
11	C10	0.0000	C	0.8345	0.3779	0.2289	sp3	0	0.04692
12	C5	0.0000	C	0.2978	0.620	0.378	sp3	0	0.0566333
13	C4	0.0000	C	0.4862	0.891	0.76	sp3	0	0.0486733
14	C11	0.0000	C	0.6496	0.1778	0.3230	sp3	0	0.0572667

OK Print

Figure 9.30 - List of parameters with Ueq to the right

Examining the values here is difficult because the atoms are not in order.

9.3.5 Sort Atoms

1. Right-click on the background and click on "Sort..." to sort the atoms into a sensible order.

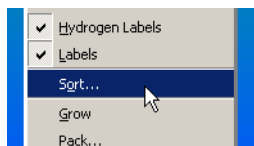


Figure 9.31 - Choose the Sort option

A box will appear to the right of the molecule display. Since S1, O1 and O2 are in a reasonable order, only the carbon atoms need to be ordered.

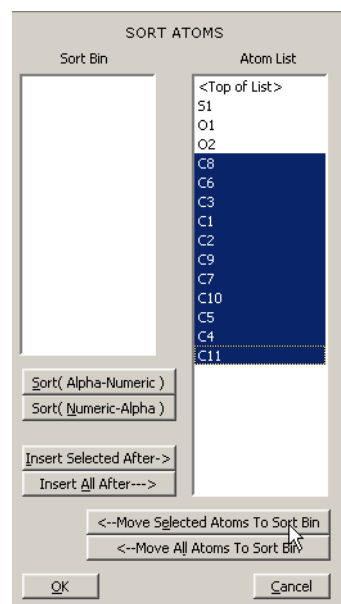


Figure 9.32 - Selecting carbons for sorting

NOTE: Generally, it is easier to sort and number atoms before adding hydrogen atoms.

2. Highlight the atoms to be sorted (i.e., left-click and drag over the atoms) and move them to the Sort Bin (i.e., left-click on Move Selected Atoms to Sort Bin).

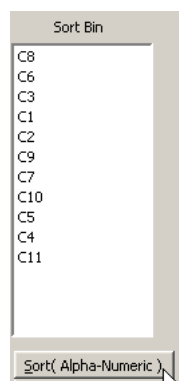


Figure 9.33 - Carbon atoms in Sort Bin

3. After the atoms are in the Sort Bin, click on Sort(Alpha-Numeric) to get them into a normal order.

4. Highlight O2 and click on Insert All After as in Figure 9.34.

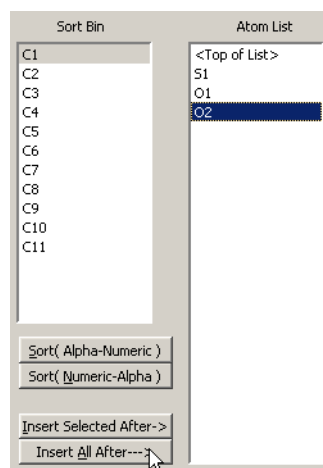


Figure 9.34 - Sorted atoms with "insert after" atom (O2) selected.

5. Click on OK to accept the sorting.



Figure 9.35 - OK button

The resulting list of atom information is easier to examine.

Information On All Atoms

INFORMATION ON ALL ATOMS									
	LABEL	PEAK HEIGHT	TYPE	X	Y	Z	HYBRID	CHARGE	UEQ
1	S1	0.0000	S	0.8097	0.1815	0.2405	sp3	0	0.0353467
2	O1	0.0000	O	0.3345	0.3026	0.1765	sp3	0	0.04722
3	O2	0.0000	O	0.8432	0.890	0.1287	sp3	0	0.0488033
4	C1	0.0000	C	0.4329	0.1949	0.1500	sp3	0	0.0335967
5	C2	0.0000	C	0.6354	0.1245	0.1726	sp3	0	0.0332767
6	C3	0.0000	C	0.6896	0.8	0.1251	sp3	0	0.03449
7	C4	0.0000	C	0.4862	0.891	0.76	sp3	0	0.0486733
8	C5	0.0000	C	0.2978	0.620	0.378	sp3	0	0.0566333
9	C6	0.0000	C	0.1496	0.502	0.228	sp3	0	0.0549167
10	C7	0.0000	C	0.1756	0.1410	0.377	sp3	0	0.0462133
11	C8	0.0000	C	0.3591	0.1141	0.827	sp3	0	0.03482
12	C9	0.0000	C	0.5093	0.24	0.679	sp3	0	0.0371967
13	C10	0.0000	C	0.8345	0.3779	0.2289	sp3	0	0.04692
14	C11	0.0000	C	0.6496	0.1778	0.3230	sp3	0	0.0572667

OK Print

Figure 9.36 - Sorted atoms

The Ueq are reasonable if there are:

- lower values for the sulfur atom and the five-member ring
- higher values for the terminal atoms—methyls and carbonyls
- sensible numbers for the six-member ring—lower for C8 and C9, higher for C5 and C6, and in between for C4 and C7.

9.3.6 Add Hydrogen Atoms

At this point, refine the hydrogen atoms either by renaming the Q peaks using the Labelling tool as before and then including them in the refinement, or include the hydrogen atoms in fixed idealized positions riding on the atom to which they are attached.

In this manual we will illustrate the latter fixed idealized method.

NOTE: In some cases (e.g., hydrogen atoms on hetero atoms or on geometrically strained carbon atoms), it is better to refine the hydrogen atom positions. A general rule is to refine if there is some doubt about the position of the hydrogen atom and there is difference electron density present.

1. Using the slider tool, remove all of the Q peaks.

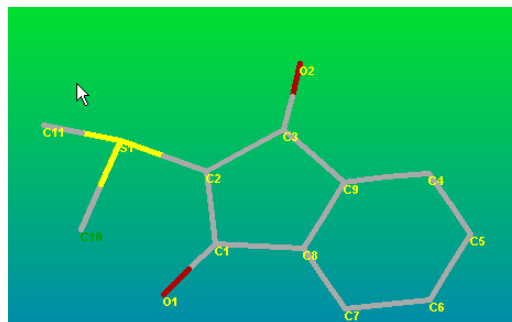


Figure 9.37 - Refined model with difference peaks removed

2. Right-click in the background and click on Hybridize All.



Figure 9.38 - Hybridize button

The colors of the atom labels will change to indicate the atom hybridization.

NOTE: If the atom hybridizations are hard to see, right-click on the background and open the Information on All Atoms window.



Figure 9.39 - Calculate Hydrogens button

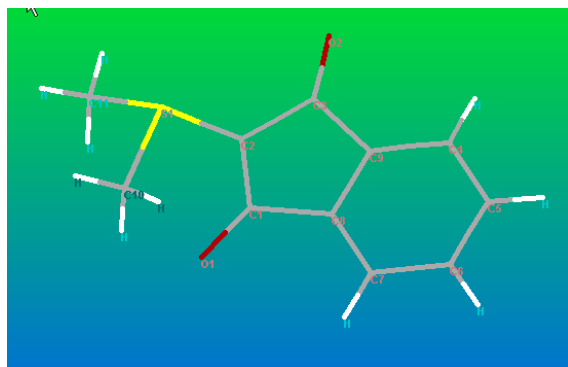


Figure 9.40 - Model with added Hydrogen atoms

3. The following warning will appear indicating that the hydrogen atoms have been provisionally added and that the final addition with naming will take place when the least-squares calculations are started. Click OK.

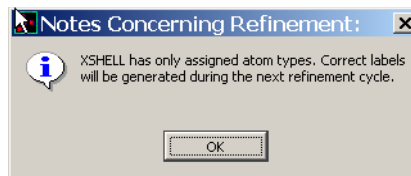


Figure 9.41 - Hydrogen atom warning

- Click on the Refine button to launch the least-squares refinement program. The output window will open and a summary of the results of individual cycles of refinement will appear.

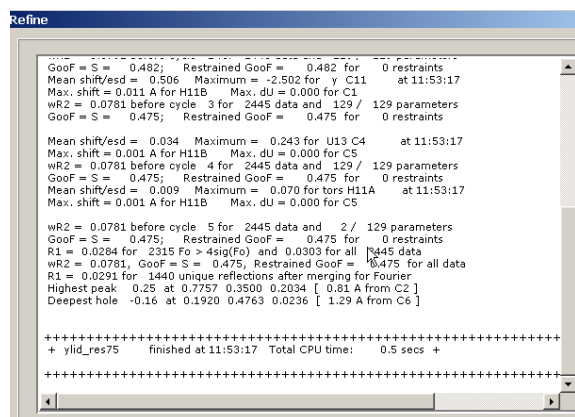


Figure 9.42 - Refinement results

The value for R1, 0.029, is typical for an anisotropic refinement with riding hydrogen atoms and a data set measured to 0.75Å. Since the mean shift/esd is less than 0.01, the model is not changing significantly. Since the highest peak and the deepest hole in the difference map are similar, there are probably no missing atoms.

The resulting difference electron density map appears rather ugly. This is because the number of difference peaks to be displayed has been left at 20.

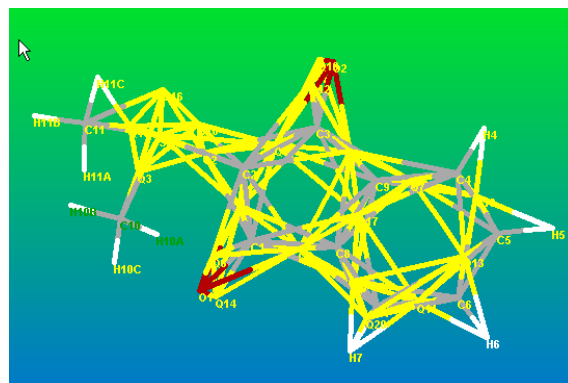


Figure 9.43 - Difference peaks in a well-refined model

9.3.7 Final Refinement for Publication

Obtain a view that is much easier to interpret by reducing the number of difference peaks in the Refine menu (see Figure 9.44).

- For the final cycles of refinement, reduce the number of peaks to be displayed to five and turn on “Use Suggested Weights” and “Generate ACTA(CIF) Information File” as shown in the next two figures.

NOTE: If the suggested weights vary significantly from 0.06 and 0.00, then change the values to 0.06 and 0.00.

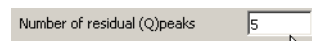


Figure 9.44 - Reduce the number of difference peaks

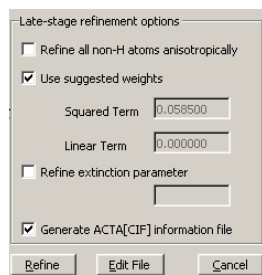


Figure 9.45 - Select weights and request ACTA output

Figure 9.46 shows a difference map with the number of peaks set at five.

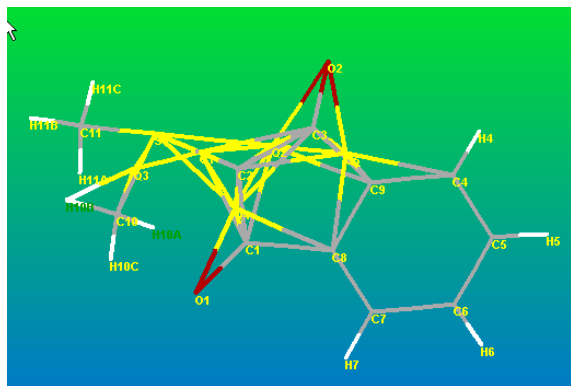


Figure 9.46 - Difference peaks

2. Click on Edit File, choose INS and click OK to open the instruction file for XL. This allows you to add instructions that are not directly available in the GUI.

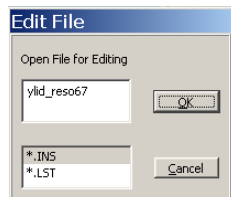


Figure 9.47 - Open the .ins file for editing

3. Check that the temperature card is correct.
4. If torsion angles are of interest, add a CONF (confirmation) card (see Figure 9.48).

```
TEMP 23
CONF
mpla 12 c1 c2 c3 c4 c5 c6 c7 c8 c9 s1 o1 o2 c10 c11
mpla 6 c4 c5 c6 c7 c8 c9 c1 c2 c3 o1 o2 s1 c10 c11
mpla 5 c8 c9 c1 c2 c3 c4 c5 c6 c7 o1 o2 s1 c10 c11
mpla 10 c1 c2 c3 c4 c5 c6 c7 c8 c9 s1 o1 o2 c10 c11
```

Figure 9.48 - Add instructions in the .ins file

5. If planarity and angles between planes are of interest, add mpla cards.

9.3.8 Generate an Atomic Displacement (Thermal Ellipsoid) Plot

1. Remove difference peaks with the slider tool.
2. Right-click on the background and select Thermal Ellipsoids.

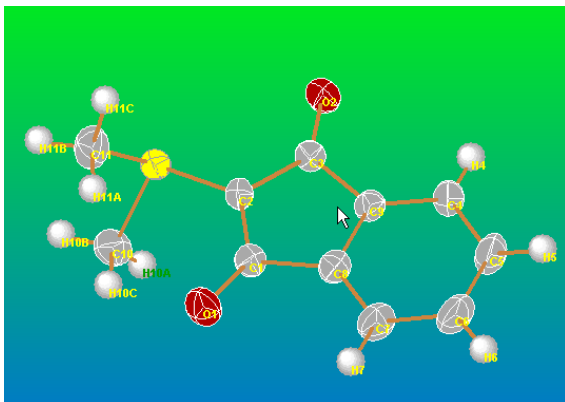


Figure 9.49 - Thermal ellipsoids with large hydrogen atoms

3. If the hydrogen atoms in Figure 9.49 are large, change their size.

- 3.1 Click on Preferences in the Window Tool Bar and choose Atom Preferences.

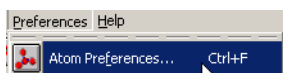


Figure 9.50 - Atom Preferences tool

At the top of the box that is opened, there are three values that can be changed to adjust the size of atoms and the bonds to them.

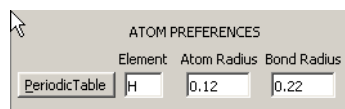


Figure 9.51 - Default atom preferences

4. Change the hydrogen radius to 0.08 and the bond radius to 0.15 to make the hydrogen atoms smaller.

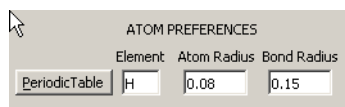


Figure 9.52 - Modified atom preferences

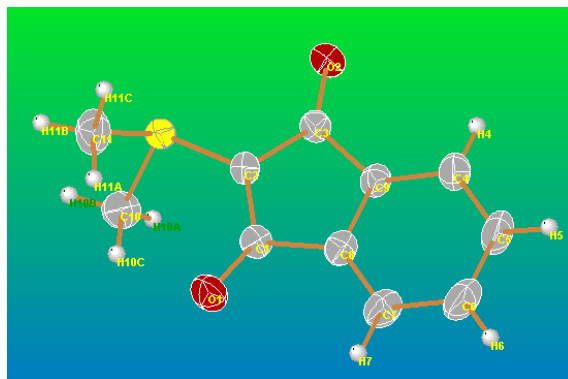


Figure 9.53 - Thermal ellipsoid plot with smaller hydrogen atoms

5. Position atom labels by right-clicking on the atom and choosing Position Label.

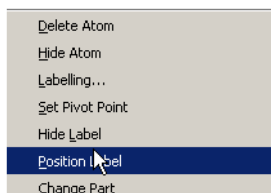


Figure 9.54 - Position Label menu

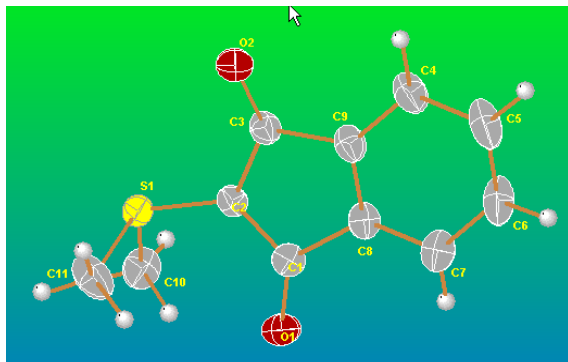


Figure 9.55 - Positioned labels

6. Convert this image into a high-quality .jpg file for inclusion into reports by clicking on render in the Windows Tool Bar and choosing Black and White to High Quality JPEG file.

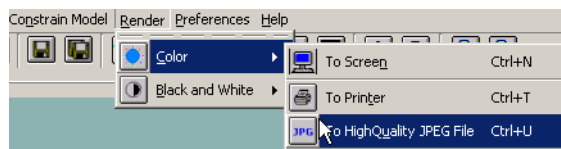


Figure 9.56 - Generate JPEG

NOTE: A message may appear that the font file needs to be defined: "Please set your font directory for the first time for opengl rendering." If this happens, click on Preferences > OpenGL Preferences. Then click on the TrueType font For.OpenGL. In Windows, browse to C:\bn\xshell\fonts\ or in Linux go to /usr/local/bin/xshell/fonts. Choose a font.

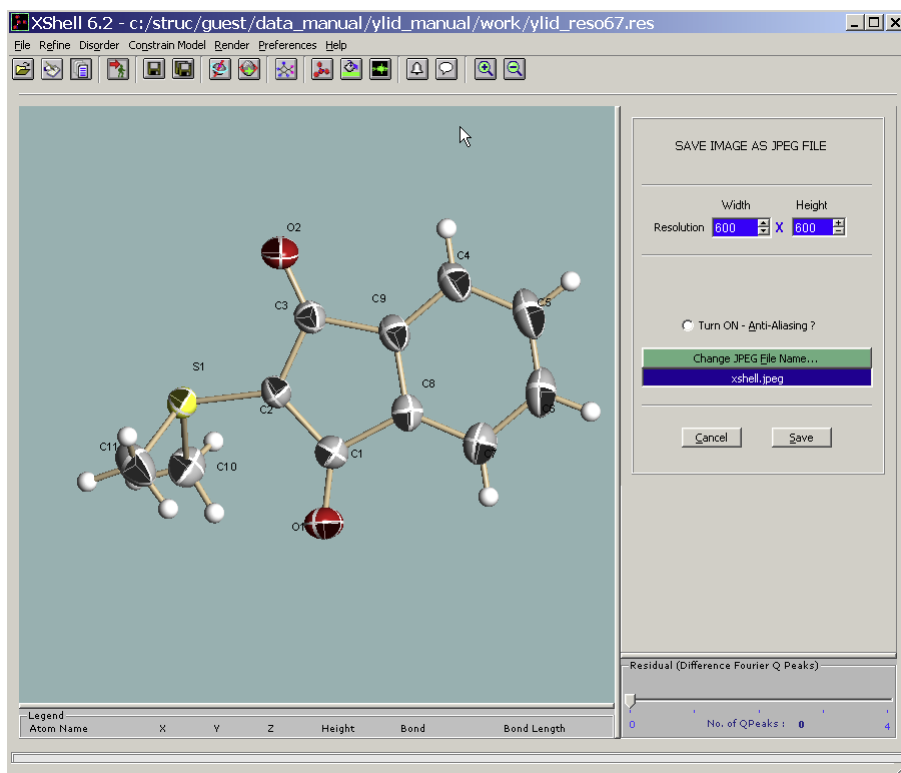


Figure 9.57 - The final rendered thermal ellipsoid/atomic displacement parameter plot

Appendix A. RLATT

RLATT is a very powerful tool for viewing the harvested reflections in a reciprocal lattice. This section will cover only a few of its features. RLATT is best learned through use.

This example has over 3300 reflections. This is not the typical case; generally only 300 or 400 reflections are examined. Using extra reflections will make the examples easier to see.

A.1 Open RLATT

1. Start RLATT from the Task Menu under "Examine Crystal."
2. Click on the Start icon to read the reflections from the database into RLATT.

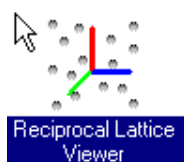


Figure A.1 - RLATT icon

Figure A.2 is a typical unoriented view after import. The gray circles are more intense reflections, i.e., those with higher $I/\sigma(I)$. The black dots represent less intense reflections. The intensity key slider at the bottom of the window can be used to change the selection criterion.

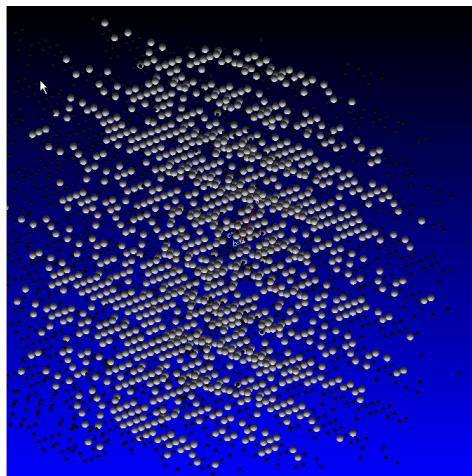


Figure A.2 - Unoriented RLATT view

Other tools can be accessed by right-clicking on the background to give the “Quick Tools” menu (Figure A.3) and by clicking on RLATT in the menu bar (Figure A.4).

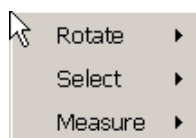


Figure A.3 - Quick Tools menu

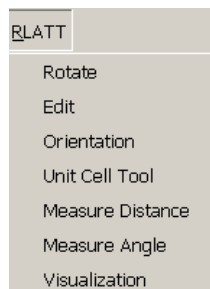


Figure A.4 - Full menu tools

Additional context-sensitive information is available by left-clicking on the “What’s This” help arrow in the menu bar.



Figure A.5 - The “What’s This” help arrow

For example, clicking on the help arrow and then clicking in the work area (the gray area to the right of the lattice display) gives the display of shortcuts shown in Figure A.6. Clicking on the blue background gives the hints shown in Figure A.7.

RLATT Keyboard Commands	
Rotate & Zoom:	
UP / DOWN	= rotate along x
LEFT / RIGHT	= rotate along y
INS / DEL	= rotate along z
+ / -	= zoom
hold SHIFT	= 4x speed
hold CTRL	= 1/4 speed
hold SHIFT + CTRL	= 16x speed
Selection Tools:	
SHIFT + left-click	= add spots to current selection
CTRL + left-click	= remove spots from current selection
UP, DOWN, LEFT, RIGHT	= move selection tool
hold SHIFT	= 4x speed (during movement only)
hold CTRL	= 1/4 speed (during movement only)
hold SHIFT + CTRL	= 16x speed (during movement only)
+	= add slice (lattice tool only)
-	= remove Slice (lattice tool only)
PGUP	= add Extension (lattice tool only)
PGDN	= remove Extension (lattice tool only)
ALT + left-click	= lock onto spot (lattice tool only)
Orientations:	
F1	= 1 0 0 (along a*)
F2	= 0 1 0 (along b*)
F3	= 0 0 1 (along c*)
F4	= unoriented (identity matrix)
F5	= user-defined orientation 1
F6	= user-defined orientation 2
F7	= user-defined orientation 3
F8	= user-defined orientation 4
SHIFT + F5	= store user-defined orientation 1
SHIFT + F6	= store user-defined orientation 2
SHIFT + F7	= store user-defined orientation 3
SHIFT + F8	= store user-defined orientation 4
Distance Measurement:	
+	= add slice
-	= remove Slice
PGUP	= add Extension
PGDN	= remove Extension
hold SHIFT	= action x 10
ALT + left-click	= lock onto spot
Angle Measurement:	
ALT + left-click	= lock onto spot (locking onto all three calculates the angle in 3D)
Misc:	
[	= adjust intensity slider
]	= adjust intensity slider
A	= change axis modes
C	= change colorization method
I	= invert selection
M	= toggle distance measurement during lattice selection tool
N	= toggle Null Resolution Mode
S	= change 2D Projection Scale mode
U	= toggle Unit Cell Tool

Figure A.6 - Shortcut keys and commands

3D Reciprocal Lattice View

In this window is the three-dimensional display of the reciprocal lattice using OpenGL for hardware rendering.

Left-click and drag to rotate the view or use a selected feature.

Double-left click on a reflection to bring up a little statistics window. Click on the statistics window to make it disappear.

Middle-click and drag to change the zoom ratio.

Spin the mouse wheel to adjust the reflection sizing.

Right-click to make a quick-menu of special features available.

Figure A.7 - Help for the 3D display obtained by clicking the "What's This" arrow on the blue display background

A.2 Orienting Views

Move the mouse to rotate the RLATT display. It is possible to easily see rows and non-fitting peaks (see Figure A.8).

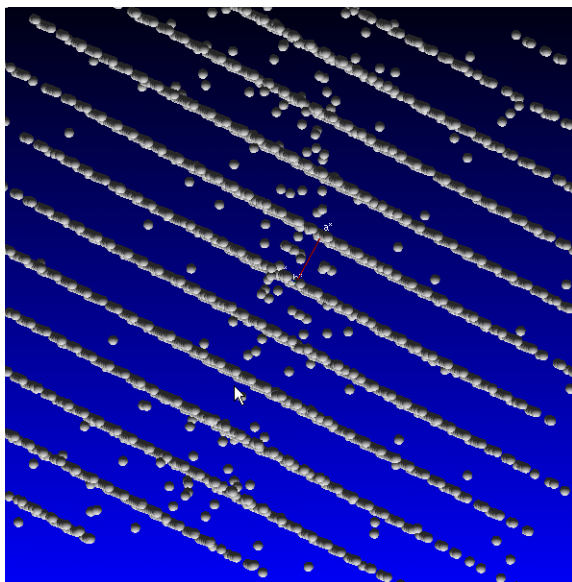


Figure A.8 - RLATT display looking down layers of reflections

By sliding the intensity key at the bottom of the display, it is easy to deselect weak reflections. In Figure A.9 the reflections between the layer lines all go black when the intensity filter is moved to the left.

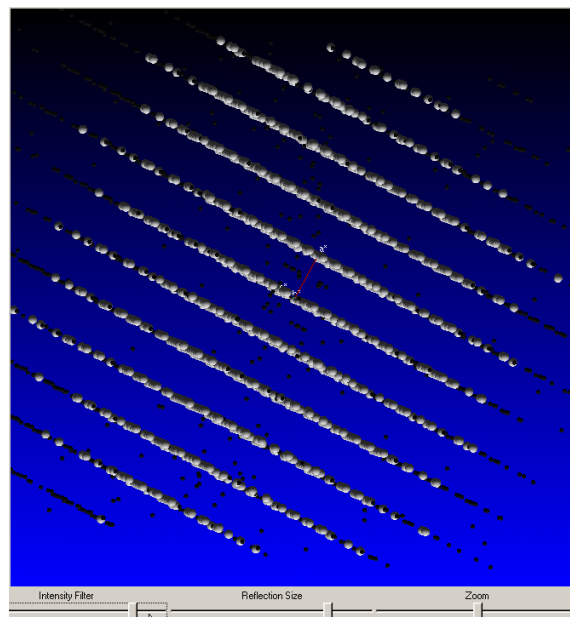


Figure A.9 - RLATT display looking down layers of reflections. Weaker reflections, i.e., those with smaller $I/\sigma(I)$, are black because the "Intensity Filter" slider (bottom left) has been moved slightly to the left.

With additional rotations, the layers of reflections can be further oriented so that stacks of reflections become visible. Sometimes it is quite useful to right click on the background and choose Z-rotations from the rotation options. The 2D profiles on the top and to the left are a valuable aid in this process. With practice, the

2D profiles can be organized into clusters. As shown, the 2D profiles are counting intensity. The counting mode can be changed to spot count or turned off completely using the Visualization menu (RLATT > Visualization) or by hitting the “s” key to cycle through the counting modes.

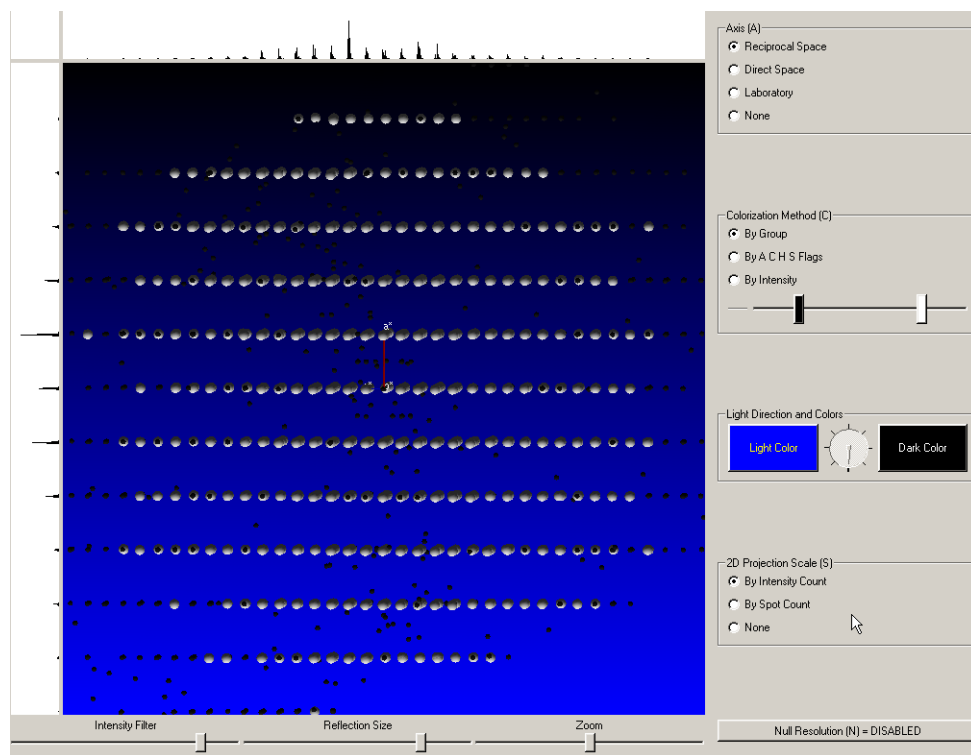


Figure A.10 - An oriented lattice view. The Visualization menu is to the right. The 2D profiles are to the left and to the top.

The distance between layers can be measured by clicking on one of the clusters of lines in the 2D view and then dragging to the next cluster. If you drag over three clusters, then the distance as measured would need to be multiplied by three.



Figure A.11 - RLATT display with measured distances in the 2D views.

The Orientation menu allows easy orientation if the cell is indexed. Hit the F1, F2, or F3 key or click on the appropriate button.

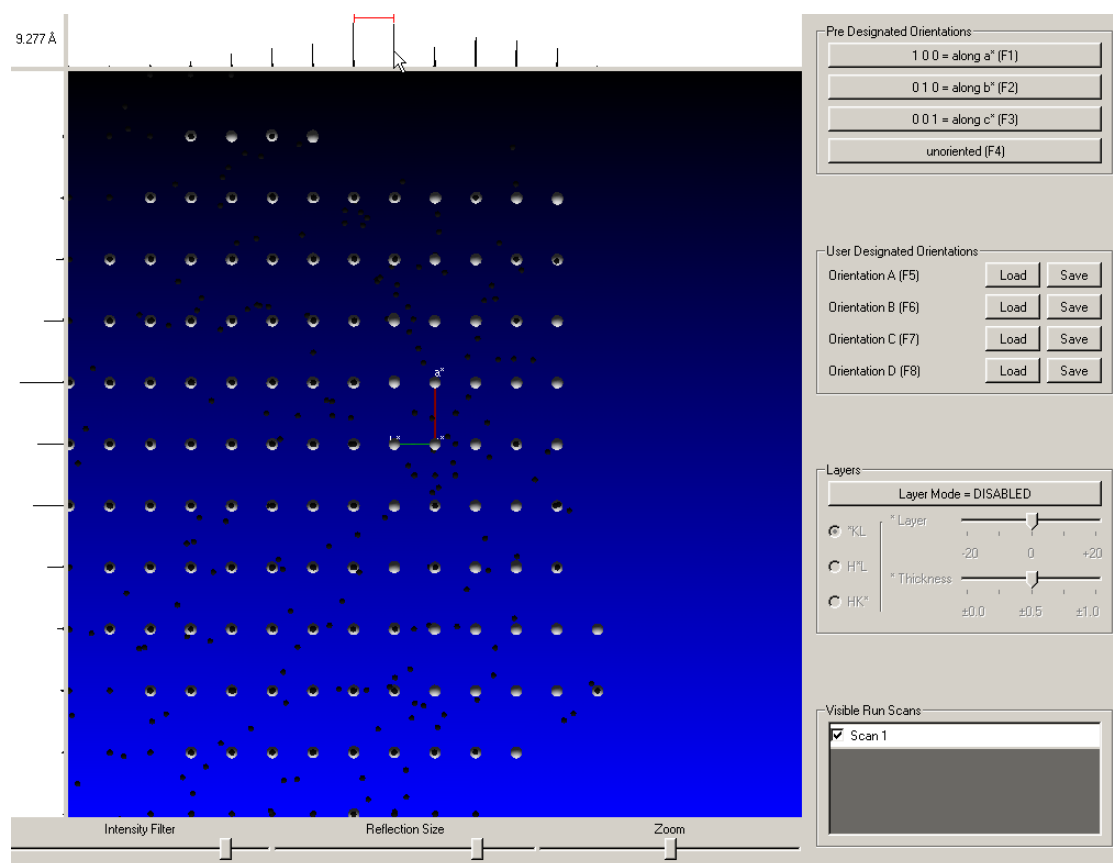


Figure A.12 - An aligned image with the Orientation menu to the left

1. Select reflections with the Lattice tool.

The Lattice tool is one of the most useful editing tools. Activate it from the Edit menu or by clicking on the background and choosing Select > Lattice. When initially activated, nothing appears. Hold down the Alt key to lock on a centroid and then left-click and hold on a spot. Now drag the mouse and line up the line that is tied to your spot on a row. Choose a longer row and align carefully. Now, while still holding the left mouse button, drag the second line to another row as shown in Figure A.13. Let go of the button. If you want to start over, click on another spot and repeat the process. Using the Alt key is optional, but it makes alignment of the lines easier and more accurate (Linux users may find that the Alt key has operating system functions).

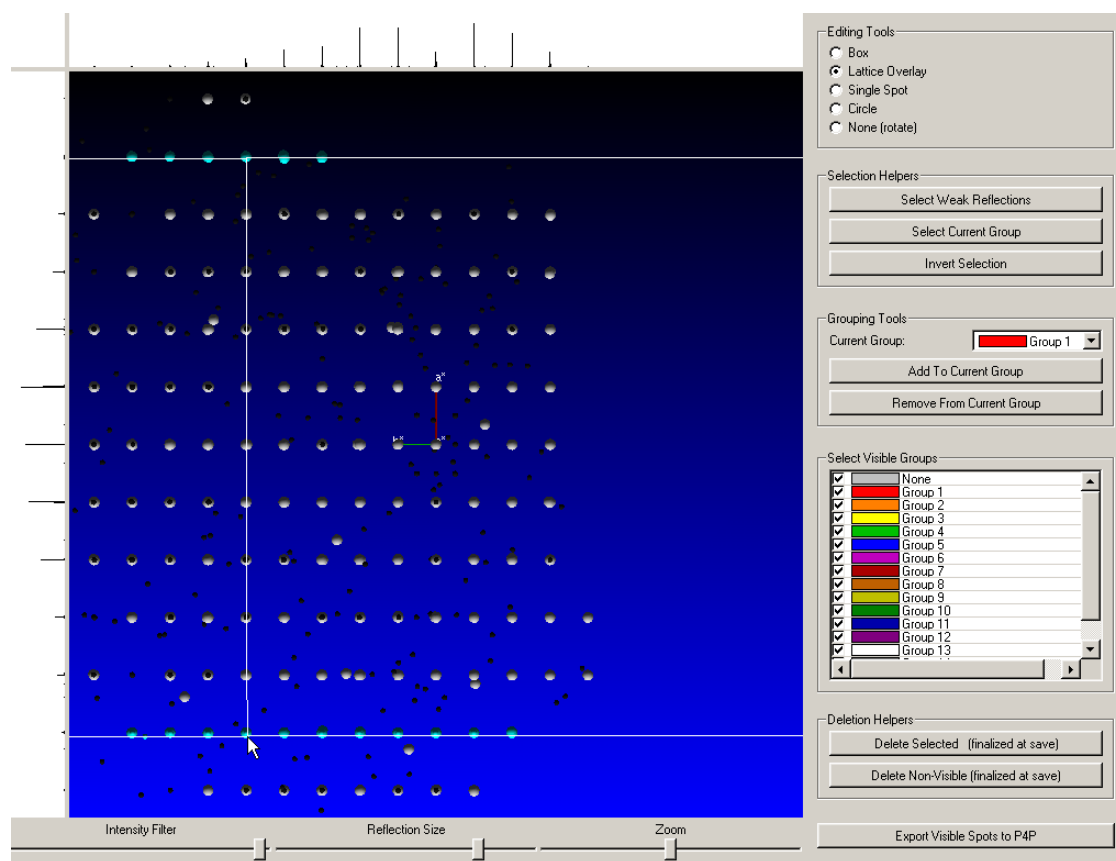


Figure A.13 - Two lattice lines selected

Hit the “plus” key to add lines between the two you have marked. Hit the “minus” key to remove lines.

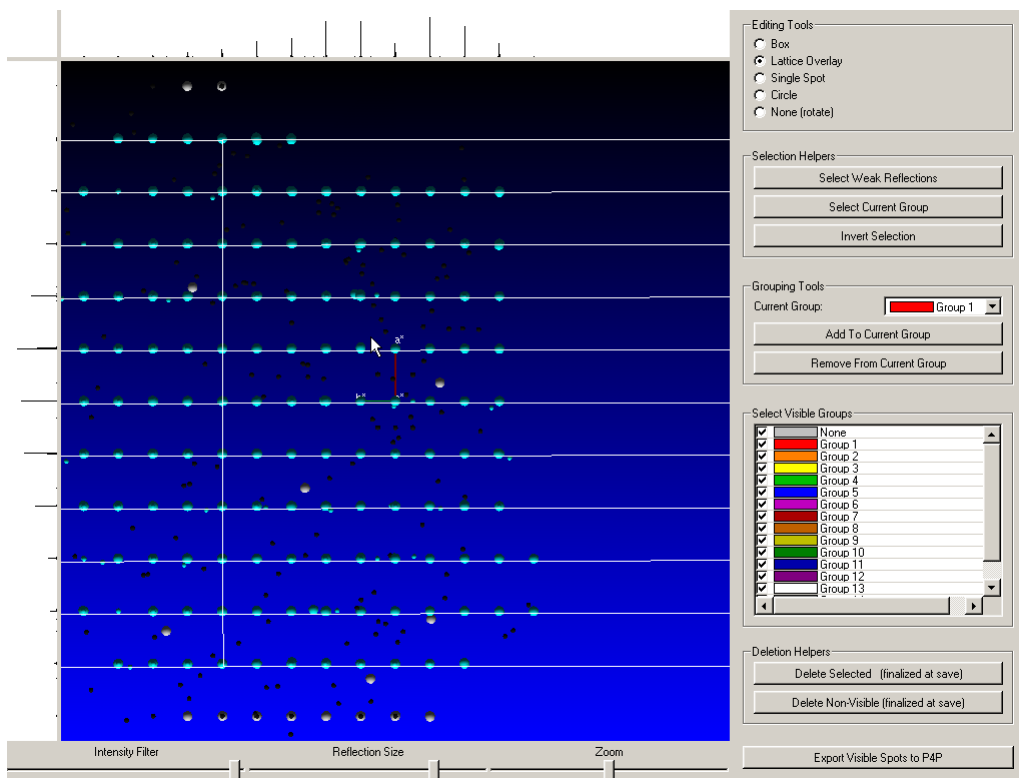


Figure A.14 - View with most of the lattice lines selected. Lines to the top and bottom are not selected.

Hit “Page Up” to add lines to the outside of the previously selected lines (in this example, the top and bottom). “Page Down” will remove lines from the outside. Now all visible lines are selected. Note that the selected reflections are turquoise.

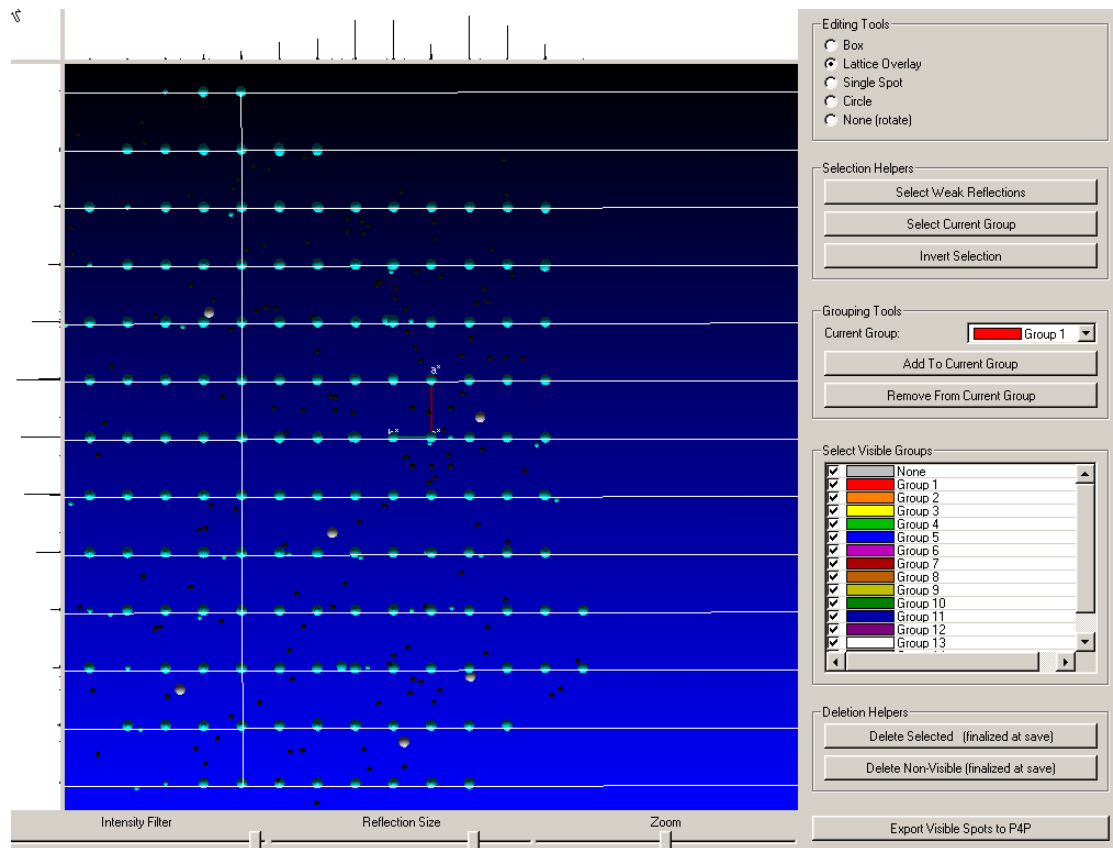


Figure A.15 - A view with all layers selected

Click on Invert Selection (in the “Editing Menu” (RLATT > Edit) under “Selection Helpers”). All of the spots that do not touch the layer lines are now selected.

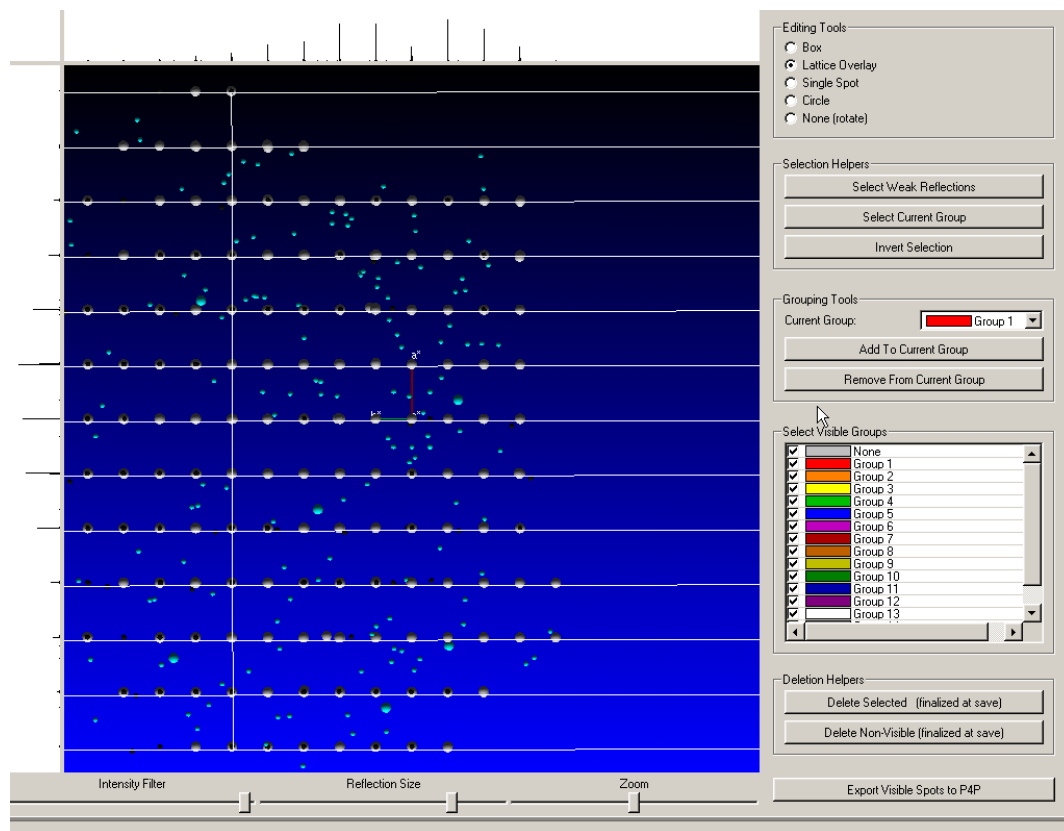


Figure A.16 - The selected spots are now the little ones between the layers. The spots in the layers are grey and the selected spots are turquoise.

Click on “Delete Selected (finalized at save)” to mark the selected reflections to be deleted (and turned black). Alternatively, these selected reflections can be added to a group and then hidden using “Select Visible Groups.”

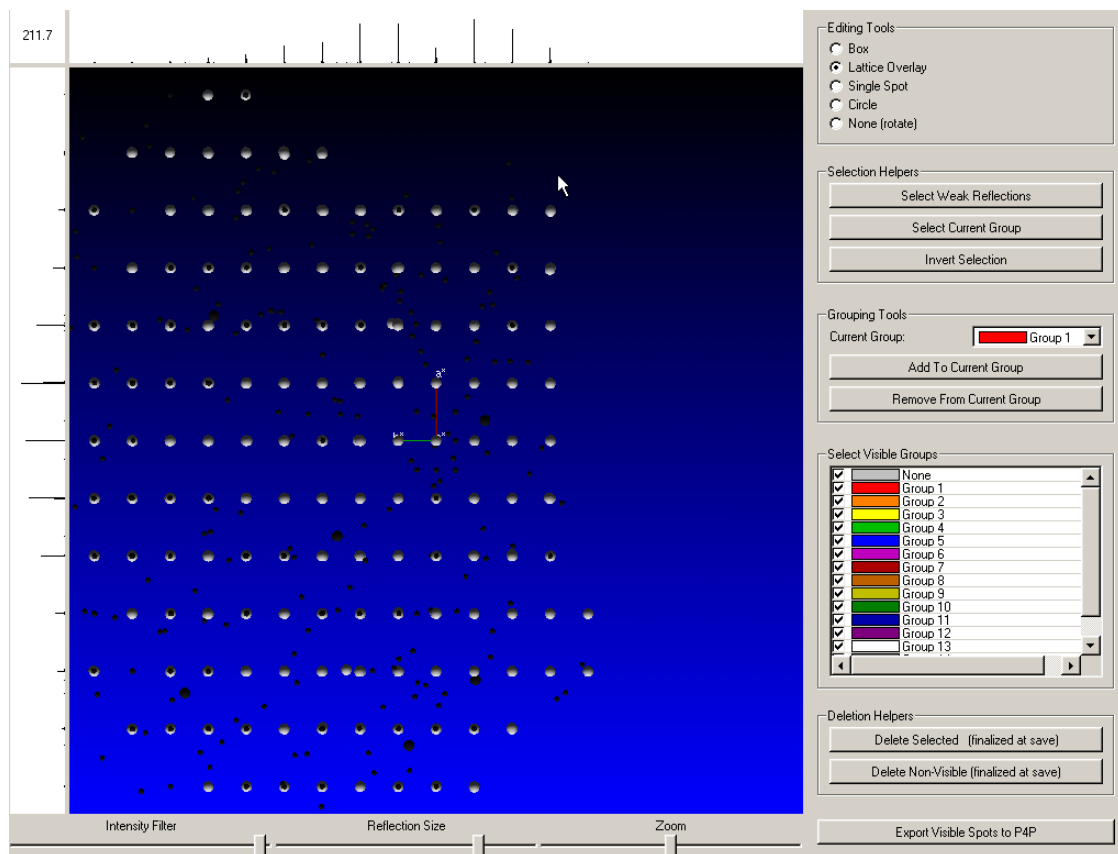


Figure A.17 - Selected reflections are blacked out

Save the file by clicking the disk icon (Figure A.18) or use File > Save to remove the reflection from the reflection list. Hiding a group with “Select Visible Groups” does not remove it from the reflection list. Either way, the resulting RLATT image is much cleaner and easier to interpret (Figure A.19).

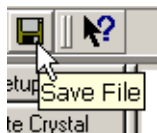


Figure A.18 - Save File icon

Enable the Unit Cell tool to put a colored box in the view. This allows you to see if reflections are actually falling on the corners of the box (see Figure A.19 and Figure A.20). There are multiple options in the Unit Cell tool. “Select a Visible Plane” displays individual planes. “Select Plane

Size” determines the boundaries of the planes and the number of unit cells displayed. Grid Planes mode displays planes as grids with spacings determined by the lattice.

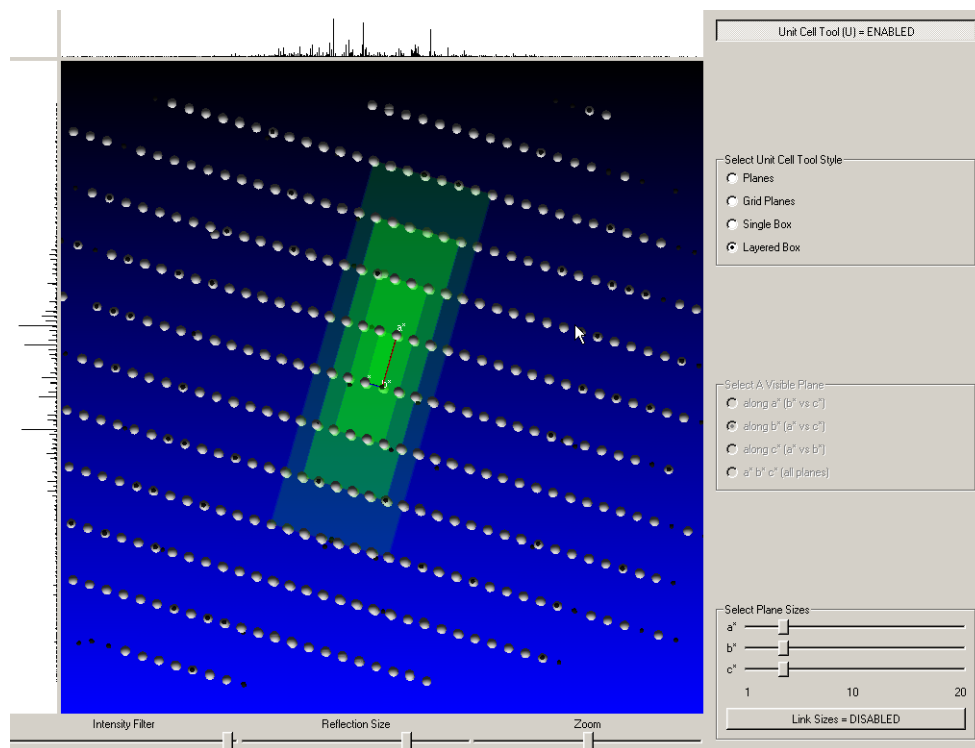


Figure A.19 - A view of the reciprocal lattice after the non-fitting reflections were deleted and with the Unit Cell tool enabled.

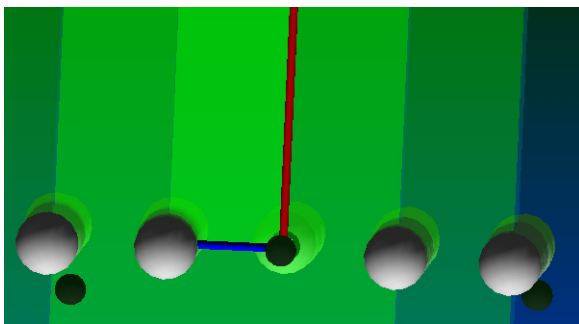


Figure A.20 - A view that zooms in on the Unit Cell tool. Almost all spots lie on the lines and planes defined by the unit cell. To the left and right are two weaker reflections (black dots) that do not fit.

A.3 Defining Groups

Selected reflections can be grouped. This tool is most useful for examining twinned, split, in-grown and otherwise problematic crystals. For Figure A.21, alternate rows were selected with the Lattice Selection tool. In the Grouping Tools section of the tool boxes to the right, the current group was set to Group 1 (red). Click on “Add to Current Group” to turn these rows red. Then, the other rows and Group 13 (white) were selected

and added. The box tool was used to select the reflection to the top left and these were put in Group 3, the yellow group. Finally, some of the reflections to the top and bottom were selected and added to Group 5, the blue group. This example of using the color groups generates a flag-type display. For twinned crystals, etc., the groups would be used to denote different components (see Figure A.22).

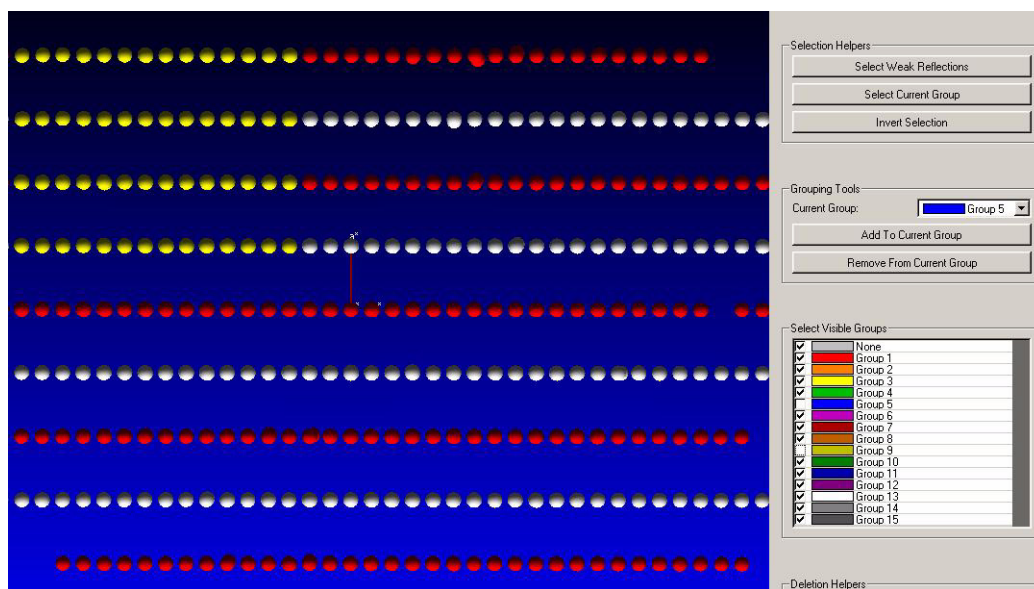


Figure A.21 - Reflections selected for different groups

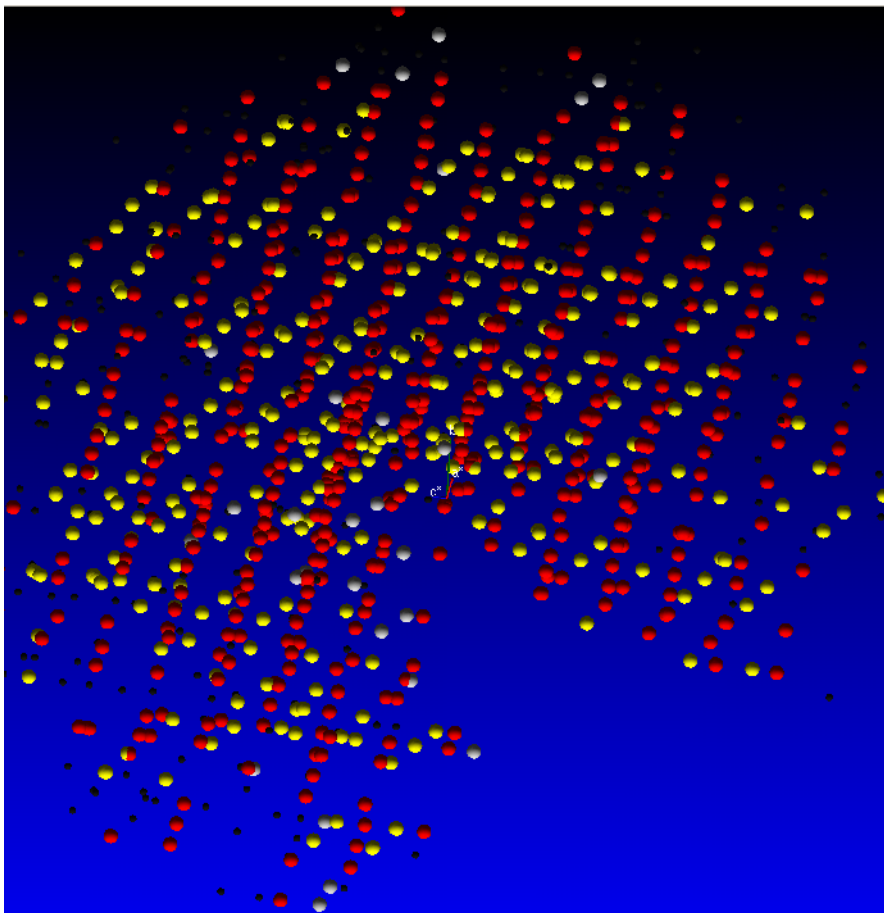


Figure A.22 - A more practical use of the selection and color groupings in RLATT. The two components of a rotational twin are colored yellow and red.

A.4 Measuring Distances and Angles

Right-click on the background and choose Measure to get tools for measuring distances and angles.

The Measure Distance tool gives two lines, much like the lines in the Lattice Selection tool, that can be oriented and dragged to get lattice spacings (see Figure A.23). Use the Alt key to lock on spot centers for easier orientation and more accurate measuring.

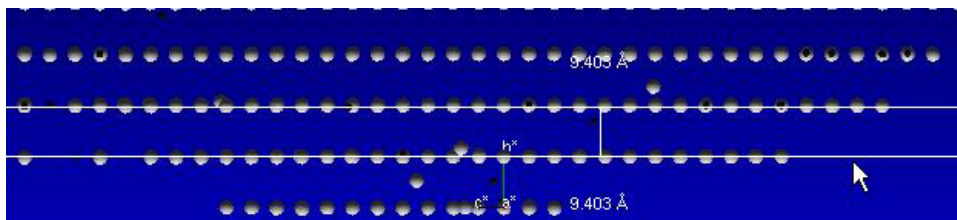


Figure A.23 - Using the Measure Distance tool

With the Measure Angle tool, left-click, hold, and drag a line on a lattice layer ending on the spot that will become the vertex of the angle. When the mouse is released, that point becomes the pivot point for a new line. Moving the mouse with no buttons depressed gives angle measurements (see Figure A.24). As described, the angle measurements will be done in 2D mode

(i.e., the angle between the two lines displayed). To measure an angle in 3D mode, hold the Alt key while selecting spots as above. The tool will then lock on to spot centroids and the angle will be calculated using spot coordinates. This eliminates errors that might result from measuring using the 2D projection of the current orientation.

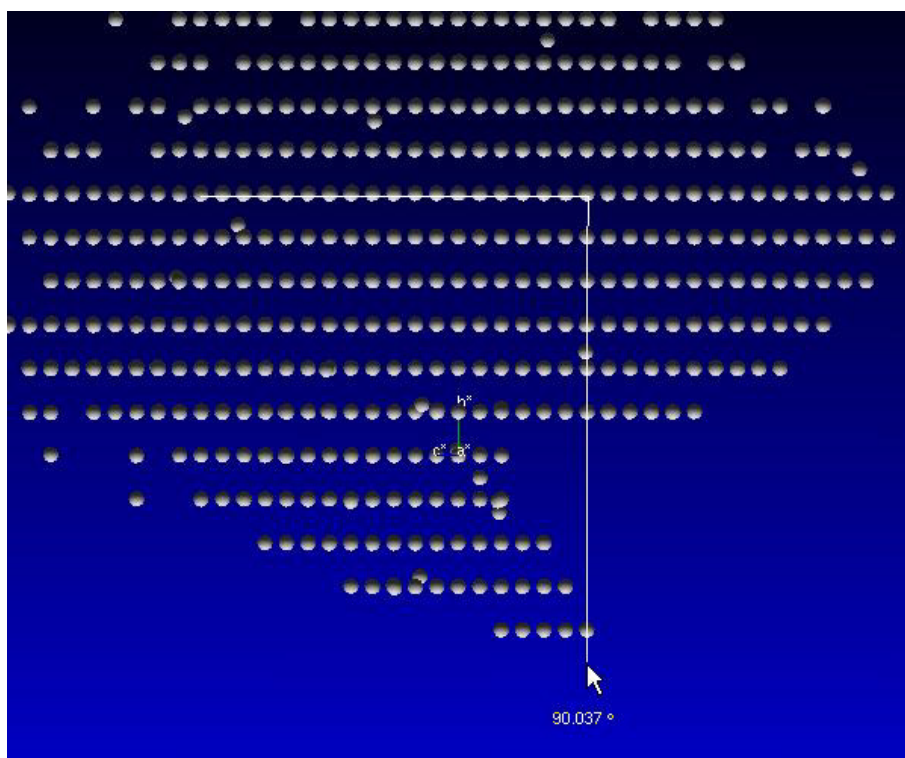


Figure A.24 - Using the Measure Angle tool

A.5 Writing a .p4p File

At the bottom of the Edit menu, there is a button for exporting all **visible** reflections to a .p4p file. The .p4p file is a text file of crystal, instrument, and reflection information. If the cell and crystal orientation is known, that information is included also (otherwise, dummy values are included). The sequence of cleaning up a set of reflections using RLATT and then exporting the results for input to CELL_NOW is a useful tool for dealing with hard-to-index crystals.

Appendix B. SaintPlus

Occasionally, it is necessary to integrate images outside of the APEX2 GUI. Usually this is necessary in order to import a matrix (.p4p) file. SaintPlus, executed from a command window, is designed for this purpose.

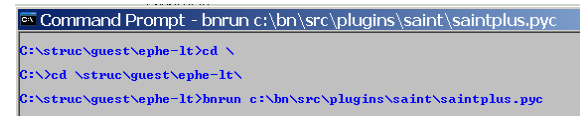
1. Open a Command Prompt window.
2. Change directories to the folder of interest.
3. For Windows, enter the command

```
bnrun c:\bn\src\plugins\saint\  
saintplus.pyc
```

(see Figure B.1).

4. For Linux, enter the command

```
bnrun /usr/local/bn/src/plugins/saint/  
saintplus.pyc.
```



```
Command Prompt - bnrun c:\bn\src\plugins\saint\saintplus.pyc  
C:\struc\guest\ephe-1t>cd \  
C:\>cd \struc\guest\ephe-1t\  
C:\struc\guest\ephe-1t>bnrun c:\bn\src\plugins\saint\saintplus.pyc
```

Figure B.1 - Starting SaintPlus

After the command is executed, the SaintPlus window will open (see Figure B.2).

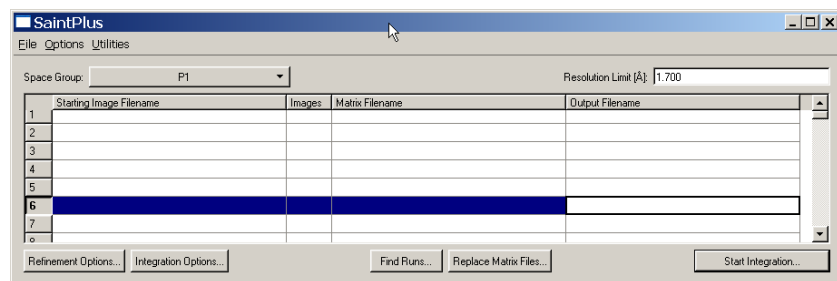


Figure B.2 - Initial integration window

This window is like the Saint window discussed previously (Section 7), but with the following differences:

- Defaults are not determined by previous knowledge of the sample.
 - There is a button to replace the matrix files.
5. Click Find Runs.
 6. Browse to find the images to integrate.

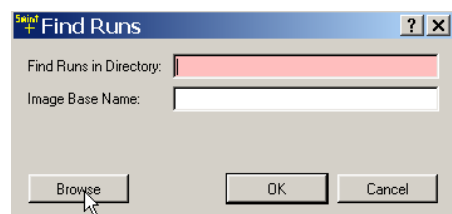


Figure B.3 - Find Runs window

7. Click on Replace Matrix Files and browse to find a .p4p or .spin file to use.

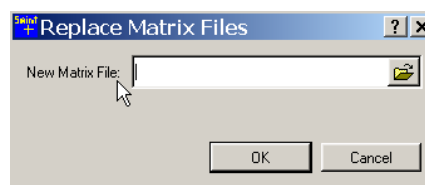


Figure B.4 - Find Matrix window

8. This defines the information on the runs, but the defaults for space group and resolution have not been updated. Click on Space Group and select the correct space group, in this case Pmmm. Actually, it is the Laue symmetry that is important here. you will note that 'Pmmm' is starred in the list, indicating a preferred choice (see Figure B.5).

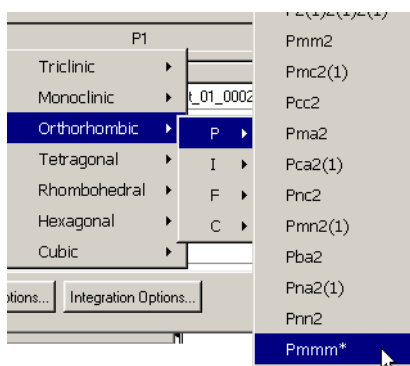


Figure B.5 - Space group tool

9. Double-click on the Resolution Limit and enter an appropriate value.

NOTE: If the starting values for SAINT need to be changed frequently, change the default by modifying the SAINT.ini file. Consult the SAINT manual 269-0175xx for further information.

10. Use the Refinement Options and Integration Options buttons to set up the SAINT run. In this case, Generate Active Mask has been turned on and set to 0.7.

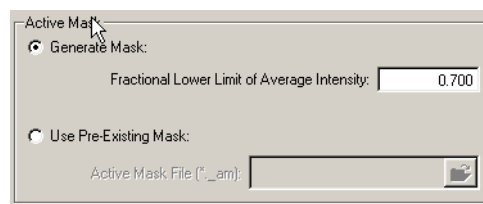


Figure B.6 - Active Mask tool

11. Click on Start Integration to start the calculations and open the SaintChart window for monitoring the results.

Appendix C. Using CELL_NOW

CELL_NOW is an extremely powerful tool for determining unit cells for difficult crystals. It analyzes a list of reflections to find a cell and orientation matrix despite the presence of several twin domains or other problems.

In initial search mode, the program tries to find sets of reciprocal lattice planes that pass close to as many reflections as possible. The corresponding real space vectors are sorted on a figure of merit (1.0 being a perfect fit). After the vector list has been output, CELL_NOW attempts to suggest a suitable cell. This will not necessarily be the conventional cell, so it should be checked using XPREP.

Once a cell is found, it may be rotated to locate further twin domains iteratively using only the reflections that have not yet been indexed.

If CELL_NOW fails to suggest a sensible cell, either something is seriously wrong with

the reflection list (e.g., a wrong detector distance) or a cell axis is longer than the given search range.

C.1 Running CELL_NOW

CELL_NOW must be run from the command line. It requires a list of reflections exported from the APEX software (a .p4p file).

1. Open a Command Prompt window.
2. Change directories to the folder containing the exported reflection list.
3. For Windows or Linux, enter the command:

```
cell_now
```

After the command is entered, the terminal will print out an explanation of the program. Read this (a copy is on the next page) as it has useful information about what the program is doing.

CELL_NOW analyses a list of reflections to find a cell and orientation matrix despite the presence of several twin domains or other junk. In initial search mode the program tries to find sets of reciprocal lattice planes that pass close to as many reflections as possible. The corresponding real space vectors are sorted on a figure of merit (1.0 would be a perfect fit). In the output these are followed by the percentages of reflections that fit within 0.1, 0.2 and 0.3 times the interplanar separation, the components a_1 , a_2 and a_3 of the vector, the angles to previous vectors and a cross figure of merit to previous vectors. The latter should be larger for reflections belonging to the same twin component. Cosines of angles between vectors a and b can also be calculated from the components by $(a_1*b_1+a_2*b_2+a_3*b_3)/(a*b)$.

After the vector list has been output, CELL_NOW attempts to suggest a suitable cell. This will not necessarily be the conventional cell, so it should be checked using XPREP (without an .hkl file) taking the lattice type found with CELL_NOW into account. If necessary this conventional cell may be reinput in 'specified cell' search mode to find the orientation matrix. If CELL_NOW fails to suggest a sensible cell, either something is seriously wrong with the reflection list (e.g. a wrong detector distance) or a cell axis is longer than the given search range.

In specified cell search mode the program tries to find the best cell within the specified ranges. The reflections that fit this cell within a specified fraction of all three interplanar spacings may be flagged as indexed, and a new .p4p or .spin file written in which they have the 'H' flag so that they can be displayed in a different color with RLATT. Then the cell may be rotated to locate further twin domains iteratively using only the reflections that have not yet been indexed.

** WARNING: the exhaustive search employed in this program is VERY SLOW **
** so a CPU clock frequency of AT LEAST 3GHz is strongly recommended **

CELL_NOW will then request the name of the input file and will suggest a name for the output file.

4. Enter the name of the input file (For the output file, hit <Enter> to accept the default).

```
Full name of .p4p, .spin or .drx file to read: example.p4p
999 reflections read in
```

```
Listing file [example._cn]:
```

```
514 reflections read in
Listing file [example._cn]:
```

5. Hit <Enter> to start a general search.

```
Initial search (<Enter>) or specified cell search (S):
```

6. If the default superlattice threshold is suitable, hit <Enter>.

```
Superlattice threshold: an axis will be rejected if less than this percentage
of reflections has indices not equal to 2n or 3n resp. [10]:
```

7. If the default minimum and maximum distances are acceptable, hit <Enter> again.

```
Minimum and maximum allowed values for cell edge [5 40]:
```

NOTE: Usually CELL_NOW is used to search generally for a cell, but if the cell is known, choose the specified cell option and enter the cell dimensions. CELL_NOW will then search for a matching cell.

After some period of time, a list of real space vectors sorted on a figure of merit (1.0 being a perfect fit) will be output. These are followed by the percentages of reflections that fit within 0.1, 0.2 and 0.3 times the interplanar separation, the components a1, a2 and a3 of the vector, the angles to previous vectors and a cross figure of merit to previous vectors. The latter should be larger for reflections belonging to the same twin component. Cosines of angles between vectors a and b can also be calculated from the components by $(a_1*b_1+a_2*b_2+a_3*b_3)/(a*b)$.

Length, fom, #hits, %(0.1), %(0.2), %(0.3), components
 Followed by angle/correlation to previous vectors:

12.692	0.902	30	94.493	99.999	99.999	-0.475	5.493	-11.432
11.016	0.891	20	99.098	99.698	99.899	0.491	-9.912	-4.782
90.0								
0.798								
16.802	0.836	24	93.793	99.398	99.698	-0.011	4.417	16.211
139.0	131.0							
0.754	0.755							
16.805	0.819	25	90.189	99.498	99.899	0.953	-15.402	6.654
139.1	49.1	81.9						
0.749	0.739	0.688						
27.693	0.741	19	80.379	93.692	98.898	-0.446	1.111	-27.668
23.4	66.6	162.4	115.7					
0.675	0.657	0.638	0.616					
27.701	0.736	43	78.477	92.091	97.296	1.396	-20.915	18.109
156.6	66.6	64.4	17.5	133.2				
0.687	0.659	0.620	0.645	0.569				
25.428	0.722	45	77.376	98.497	99.598	-0.496	14.332	20.999
119.9	150.1	19.1	101.0	143.3	83.5			
0.651	0.674	0.638	0.600	0.557	0.533			
18.381	0.688	35	72.271	86.285	91.791	-15.128	9.256	4.827
89.3	127.1	67.3	113.8	103.2	104.5	58.9		
0.596	0.605	0.556	0.544	0.480	0.484	0.487		
6.329	0.686	4	65.465	65.465	65.465	0.248	-2.729	5.705

```

179.9  90.1  40.9  41.0 156.7  23.5  60.0  90.7
0.574  0.573  0.532  0.524  0.459  0.469  0.465  0.426

    14.648  0.684    18    73.873  85.484  91.090  -14.633  -0.652    0.025
    89.1  90.3  90.5  90.9  89.3  90.9  90.2  36.8  91.1
    0.591  0.603  0.556  0.539  0.475  0.477  0.490  0.539  0.424
.
.
.
-----

```

After the vector list has been output, CELL_NOW attempts to suggest a suitable cell. This will not necessarily be the conventional cell, so it should be checked using XPREP (without an .hkl file) taking the lattice type found with CELL_NOW into account. If necessary, this conventional cell may then be input in 'specified cell' search mode to find the orientation matrix.

The following cells would appear to be plausible, but should be checked using XPREP because they are not necessarily the conventional cells.

FOM, % within 0.2, a..gamma, volume and lattice type for potential unit-cells:

```

    1 1.000  85.3   12.692  11.016  14.648   89.70   90.95   90.00   2047.7  P
-----

```

Cell for domain 1: 12.692 11.016 14.648 89.70 90.95 90.00

Figure of merit: 0.619 %(0.1): 68.3 %(0.2): 85.3 %(0.3): 91.0

CELL_NOW has identified a domain that fits 85.3% of the data with a tolerance of 0.2.

The output shows a suitable cell with its orientation matrix, figure of merit and the deviations of the input reflections from integer indices. This deviation is used to determine which reflections fit this cell and which are left for further searches for new domains.

```
Orientation matrix:  0.00164338 -0.00357404 -0.06816319
                   -0.03417112  0.08170117 -0.00385047
                   0.07098872  0.03940338  0.00098036
```

Maximum deviation from integer index [0.25]:

8. Enter the maximum deviation from integer index cutoff (Typically the default is reasonable). Hit <Enter>.

Percentages of reflections in this domain not consistent with lattice types:

A: 50.1, B: 49.9, C: 45.0, I: 48.9, F: 72.5, O: 64.3 and R: 65.8%

Lattice information (given as % of relevant reflections that do not fit a particular lattice) should be used in XPREP to determine the conventional cell. Typically, for a lattice to be present the % inconsistent should be less than 10, but problem crystals can make the value higher.

Percentages of reflections in this domain that do not have:

h=2n: 36.3, k=2n: 49.8, l=2n: 49.3, h=3n: 69.1, k=3n: 67.2, l=3n: 66.4%

This percentage information is useful for identifying supercell problems. Typically, the percentages should be 30% or higher for the 2n values and 50% or higher for the 3n values, but problem crystals or crystals with heavy atoms in special positions can make the values lower. Low values for a particular class of reflections can suggest that an axis is double (2n) or triple (3n) its correct length.

New cell from list (number), reorientate (R), accept (A) or quit (Q) [A]: A

9. Enter A or just hit <Enter> to accept this cell. The program will ask for a name for a .p4p file. Enter the name of the .p4p file for this solution (in this example, domain1.p4p).

.p4p or .spin file to write domain to: domain1.p4p

RLATT color-coding employed in file: domain1.p4p

White: indexed for first domain

Red: not yet indexed

877 reflections within 0.250 of an integer index assigned to domain 1,
877 of them exclusively; 122 reflections not yet assigned to a domain

122 reflections, approximately 1/10 of the data, have not yet been indexed (assigned to a domain). This is more than might be expected for random noise peaks. A search for an additional domain makes sense. The domain 1 cell will be rotated to locate further twin domains iteratively using only the reflections that have not yet been indexed.

Re-refine initial cell (R), search for next domain (S), quit (Q) or choose
new cell from list (enter number) [S]: S

10. Enter S to search for another orientation of the newly chosen cell.

Cell for domain 2: 12.692 11.016 14.648 89.70 90.95 90.00

Figure of merit: 0.643 %(0.1): 69.7 %(0.2): 97.5 %(0.3): 98.4

Orientation matrix: 0.00598966 0.00402327 0.06807401
-0.03367313 -0.08180340 0.00480114
0.07099302 -0.03914713 -0.00221035

Rotated from first domain by 179.7 degrees about
reciprocal axis 1.000 0.001 -0.032 and real axis 1.000 0.002 -0.010

Twin law to convert hkl from first to 0.999 0.004 -0.019
this domain (SHELXL TWIN matrix): 0.003 -1.000 -0.004
-0.064 0.006 -0.999

CELL_NOW has identified an additional domain that fits 97.5% of the data with a tolerance of 0.2. The relationship between this domain and the first domain and the SHELXL TWIN matrix are reported. This example is for a rotational twin about the a^* axis.

The output shows a suitable cell with its orientation matrix, figure of merit and the deviations of the input reflections from integer indices. This deviation is used to determine which reflections fit this cell and which are left for further searches for new domains.

11. Enter the maximum deviation from integer index cutoff (typically the default is reasonable).

```
Maximum deviation from integer index [0.25]:
```

12. Enter the name of the .p4p file for this solution (in this example domain2.p4p).

```
.p4p or .spin file to write domain to: domain2.p4p
```

```
RLATT color-coding employed in file: domain2.p4p
White: indexed for first domain
Green: current domain (but not in a previous domain)
Red: not yet indexed
```

```
691 reflections within 0.250 of an integer index assigned to domain 2,
119 of them exclusively;      3 reflections not yet assigned to a domain
```

```
Re-refine initial cell (R), search for next domain (S), quit (Q) or choose
new cell from list (enter number) [Q]:
```

13. Enter <q> to exit CELL_NOW.

```
C:\struc\guest\twin>
```

NOTE: At this point CELL_NOW has indexed a two-component twin. The orientation matrices for the two components are in the domain2.p4p file discussed below. Typically the next step in processing is to integrate using SAINTPLUS with the multi-component p4p file, in this case domain2.p4p, used as the matrix file.

C.2 CELL_NOW output

In this example, in addition to the terminal output described above and the example.cn listing file written to disk, CELL_NOW has written two .p4p files, domain1.p4p and domain2.p4p. The second of these is a multicomponent .p4p for a two component twin. It is shown below.

The following entries appear twice – once for each domain.

```
CELL, ORT1, ORT2, ORT3, ZEROES, ADCOR
CELL2, ORT12, ORT22, ORT32, ZEROES2, ADCOR2
```

This will eventually trigger SAINT to treat this data set as a twin.

```
FILEID SAINT          V6.43A          4.00          06/19/03 09:36:24 mraa
SITEID RufM                                Ruf
TITLE  [RCpCrCl2.nTHF]n
CHEM   C40H58Cr2Cl4O2
CELL    12.6918   11.0161   14.6480   89.7011   90.9494   90.0010   2047.695
CELLSD   0.0025   0.0022   0.0029   0.0300   0.0300   0.0300   1.024
ORT1      0.001643376   -0.003574040   -0.068163186
ORT2      -0.034171116   0.081701167   -0.003850471
ORT3      0.070988722   0.039403379   0.000980361
ZEROS    0.0000000   0.3249754  -0.0041494   0.0000   0.0000   0.0000
ADCOR      2.9302   -0.0047   0.0003   -0.8959   0.0123   0.0937
CELL2    12.6918   11.0161   14.6480   89.7011   90.9494   90.0010   2047.695
CELLSD2   0.0025   0.0022   0.0029   0.0300   0.0300   0.0300   1.024
ORT12     0.005989663   0.004023272   0.068074010
ORT22     -0.033673126  -0.081803404   0.004801138
ORT32     0.070993021  -0.039147127  -0.002210349
ZEROS2    0.0000000   0.3249754  -0.0041494   0.0000   0.0000   0.0000
ADCOR2     2.9302   -0.0047   0.0003   -0.8959   0.0123   0.0937
SOURCE Mo    0.71073   0.70930   0.71359   2.00000   50.00   40.0
```

Appendix D. Processing Twinned Data

D.1 Integration with SAINTPLUS

The multicomponent .p4p file written by cell_now is processed using SAINTPLUS.

1. Open a Command Prompt window.
2. Change directories to the folder containing the exported reflection list.
3. Depending on your operating system, enter the command to start SAINTPLUS:

Windows:

```
bnrun c:\bn\src\plugins\saint\  
saintplus.pyc
```

Linux:

```
bnrun /usr/local/bn/src/plugins/saint/  
saintplus.pyc
```

After the command is entered, the SAINTPLUS GUI will open.

4. Use of SAINTPLUS is described in appendix A.
5. The output from a multicomponent integration is a .mul file rather than the normal .raw file. This file will be processed with TWIN-ABS.

D.2 Scaling with TWINABS

1. In the same command window, change directories to the work directory and enter the command TWINABS.

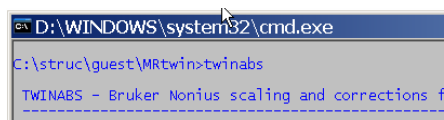


Figure D.1 - Starting TWINABS at the command prompt

2. Set the number of reflections to process and the output file. The defaults are usually correct.

```
TWINABS - Bruker Nonius scaling and corrections for twinned crystals - V1.05
```

```
-----
Maximum number of reflections allowed (500000):
Enter listing filename [twin.abs]:
```

3. Check the Laue group carefully. It always defaults to 2 and is often incorrect. In this case, the unit cell is monoclinic and the correct answer is 2.

Laue group numbers:

[1] -1	[8] -3m (rhombohedral axes)
[2] 2/m (Y unique)	[9] -31m (Z unique)
[3] mmm	[10] -3m1 (Z unique)
[4] 4/m (Z unique)	[11] 6/m (Z unique)
[5] 4/mmm (Z unique)	[12] 6/mmm (Z unique)
[6] -3 (rhombohedral axes)	[13] m3
[7] -3 (Z unique)	[14] m3m

```
[0] to write list of equivalent indices for Laue/point groups to listing file
Enter Laue group number [2]: 2
```

4. The default values for the next two inputs are usually correct.

Treat Friedel opposites as equivalent for parameter refinement (Y or N)?
Answering "N" halves the data to parameter ratio and is not recommended
unless you have a high redundancy and know what you are doing [Y]:

5. Supply the name of the data file to be scaled. Often this is a 0m.mul file. In this case, the file is 'twin.mul'.

Read reflection files written by EVALCCD with extension .sam specified) or by SAINT (extension .mul, default if no extension). Either individual files for each scan or a single merged file may be read. It is important that all files are from the same crystal and that reflections have been indexed consistently, i.e. that the orientation matrices are similar (no rows with signs reversed)!

Enter filename (/ if no more) []: **twin.mul**

2 twin components present

Mean and maximum errors in direction cosine check function = 0.000 0.002
The mean error should not exceed 0.008, and is usually caused by matrix changes during data processing.

Maximum 2-theta = 56.63 deg. Approximate wavelength = 0.71066 Angstroms

6. Model the systematic errors. Check the defaults. They are usually correct, but in some cases the error model can be improved by changing some or all of the defaults.

=====

PART 1 - Refinement of parameters to model systematic errors

```
9701 data ( 3680 unique ) involve component 1 only, mean I/sigma 2.1
9653 data ( 3649 unique ) involve component 2 only, mean I/sigma 2.4
5479 data ( 2500 unique ) involve 2 components, mean I/sigma 3.3
```

- 6a. Set the treatment of twin components. With limited data, as in this example, “-2” is recommended, but given more data, “0” is often used.

Enter N to fit component N and apply to all data, e.g. when one component dominates; -N for one parameter set to fit equivalent singles and composites that contain at least one contribution from domains 1..N; or 0 to fit each component separately [-2]:-2

- 6b. Set the cutoffs and initial weighting scheme.

Enter mean(I/sigma) threshold (must be positive) [3]:
 Highest resolution for parameter refinement [0.1]:
 Factor g for initial weighting scheme $w = 1/(\sigma^2(I) + (g\langle I \rangle)^2)$, where $\sigma(I)$ is estimated by SAINT and $\langle I \rangle$ is mean intensity [0.04]:

- 6c. Set up the treatment of weak, moderate or strong absorbers.

The following restraint esd should be increased for strong absorbers.
 Restraint esd for equal consecutive scale factors [0.005]:
 Suitable spherical harmonic orders are 4,1 for weak absorption and 8,5 for strong. Highest even order for spherical harmonics (0,2,4,6 or 8) [6]:
 Highest odd order for spherical harmonics (0,1,3,5 or 7) [3]:

- 6d. For fast computers, increase the number of refinement cycles.

Number of refinement cycles [15]: **30**

Refinement of a single parameter set to fit all single and composite reflections that contain at least one contribution from domains 1.. 2

 4150 single and 1483 overlapped reflections used for parameter fitting
 Effective data to parameter ratio = 2.69

R(int) = 0.0532 (selected reflections only, before parameter refinement)

Cycle	R(incid)	R(diffraction)	Mean wt.
1	0.0517	0.0504	0.9393
2	0.0496	0.0496	0.9417
3	0.0495	0.0496	0.9419
4	0.0495	0.0496	0.9420
5	0.0496	0.0496	0.9420
.			
.			
.			
29	0.0497	0.0497	0.9419
30	0.0497	0.0497	0.9419

R(int) = 0.0497 (selected reflections only, after parameter refinement)

7. Examine the results and accept them if they are reasonable (i.e., if the Rint is lowered and the mean weight has increased to approximately 1).

Repeat parameter refinement (R) or accept (A) [A]:

8. Reject reflections that don't fit (outliers) and establish the error model. The defaults are usually acceptable.

=====

PART 2 - Reject outliers and establish error model

Rejected reflections are ignored in the statistics and Postscript plots (except the detector diagnostics) and in the output .hkl files. All statistics and error estimates refer to total intensities (of single or composite reflections) only. Before applying rejections there are:

24833 total and 9829 unique reflections assuming Friedel's law.

8a. Input the resolution and rejection criteria. Typically the defaults are fine.

High resolution limit [0.1]:

|I-<I>|/su ratio for rejection [4.0]:

8b. Input the weighting terms to use in determining rejection sigmas.

g-value for use in: $su^2 = \sigma^2 + (g\langle I \rangle)^2$ ($\sigma(I)$ from SAINT).

This is only used for rejections, not for final $\sigma(I)$ values [0.04000]:

24832 total and 9829 unique reflections left after |I-<I>|/su test

9. Examine the results. They are acceptable if the number of rejected reflections is reasonable. Typically it is reasonable to reject less than half of your data. In this case there were 9829 unique reflections before applying rejections, and the same number after.

Repeat parameter refinement (P), repeat rejections (R) or accept (A) [A]:

10. If the suggested g value is reasonable (0.02 to 0.09), accept the suggested value.

g = 0.0610 gives best error model.

Enter new value for g or <CR> to accept:

11. Examine the statistics for the twin components, comparing the R(int) values for various runs and components. In general, each class of statistics should stay about the same for all of the runs and components. Accept if they are reasonable.

Statistics for singles for twin component 1

Run	2-theta	R(int)	Incid. factors	Diffrr. factors	K	Total	I>2sig(I)
1	-28.0	0.1755	0.907 - 1.005	1.002 - 1.127	0.653	2753	1024
2	-28.0	0.1471	0.962 - 1.026	0.993 - 1.126	0.629	4223	1716
3	-28.0	0.1750	0.968 - 1.062	1.014 - 1.128	0.684	1827	732
4	-28.0	0.2466	0.964 - 1.012	0.967 - 1.172	0.662	898	340

Statistics for singles for twin component 2

Run	2-theta	R(int)	Incid. factors	Diffrr. factors	K	Total	I>2sig(I)
1	-28.0	0.1522	0.907 - 1.005	1.002 - 1.129	0.632	2666	1102
2	-28.0	0.1249	0.962 - 1.026	1.000 - 1.126	0.608	4223	1855
3	-28.0	0.1447	0.968 - 1.062	1.014 - 1.128	0.658	1857	893
4	-28.0	0.1828	0.964 - 1.012	0.966 - 1.172	0.677	906	376

Statistics for all composite reflections

Run	2-theta	R(int)	Incid. factors	Diffrr. factors	K	Total	I>2sig(I)
1	-28.0	0.1018	0.907 - 1.005	1.005 - 1.124	0.611	1955	1058
2	-28.0	0.1121	0.962 - 1.026	0.986 - 1.125	0.632	2343	1221
3	-28.0	0.1184	0.968 - 1.062	1.011 - 1.096	0.617	577	251
4	-28.0	0.0750	0.965 - 1.012	0.946 - 1.110	0.706	604	402

Statistics for all single and composite reflections

Run	2-theta	R(int)	Incid. factors	Diffrr. factors	K	Total	I>2sig(I)
1	-28.0	0.1381	0.907 - 1.005	1.002 - 1.129	0.636	7374	3179
2	-28.0	0.1274	0.962 - 1.026	0.986 - 1.126	0.621	10789	4796
3	-28.0	0.1500	0.968 - 1.062	1.011 - 1.128	0.664	4261	1880
4	-28.0	0.1378	0.964 - 1.012	0.946 - 1.172	0.679	2408	1123

$su = K * \text{Sqrt}[\sigma^2(I) + (g\langle I \rangle)^2]$ where $\sigma(I)$ is estimated by SAINT

The above statistics are based on all non-rejected data, ignoring reflections without equivalents when estimating R(int) and K.

Repeat parameter refinement (P), repeat rejections (R) or accept (A) [A]:

12. Output the diagnostics plots and look at them. The plots have the same information content and meaning as in the scaling section of the main manual. The defaults are usually fine.

PART 3 - Output Postscript diagnostics and corrected data

12a. Set up the diagnostic output. Typically the defaults are fine.

Write Postscript diagnostic file (Y or N) [Y]:

Enter name of Postscript file [twin.eps]:

Short (<21 chars) title for Postscript plots [Test]:

Spatial display of $(I - \langle I \rangle) / su$ greater than [3.0] (0 for none):

13. Examination of the diagnostic plots is recommended before writing the .hkl data files.

13a. Output an HKLF 4 format file for solving the structure.

It is now possible to generate a HKLF 4 format file with crude intensities for structure solution or a more accurate HKLF 5 format file for refinement.

Repeat (R), write .hkl file (4 or 5), or quit (Q) [4]:

Average equivalent reflections (Y or N) [Y]:

13b. Change the output file to reflect that this is a HKLF 4 type file. For example, name it "hklf4" or "twin_4". The extension .hkl is added automatically.

Enter name of output .hkl file [twin.hkl]: **twin_4**

13c. Enter spherical absorption and Friedel averaging.

Mu*r of equivalent sphere for additional spherical absorption correction.

Enter <CR> if none:

Average Friedel opposites in output file (Y or N) [Y]:

13d. IMPORTANT! Specify that only component 1 should be used for the HKLF 4 file.

Use component N only (recommended); 0 to use all [1]:

5158 Corrected reflections written to file twin_4.hkl

Reflections merged according to point-group 2/m

Ratio of minimum to maximum apparent transmission: 0.807218

13e. Output an HKLF 5 format file for refining the structure (Note: TWINABS has automatically changed the default to type 5).

It is now possible to generate a HKLF 4 format file with crude intensities for structure solution or a more accurate HKLF 5 format file for refinement.

Repeat (R), write .hkl file (4 or 5), or quit (Q) [5]:

Average equivalent reflections (Y or N) [Y]:

13f. Set the output file to reflect that this is a HKLF 5 type file. For example, name it "hklf5" or "twin_5".

Enter name of output .hkl file [twin.hkl]: **twin_5**

Mu*r of equivalent sphere for additional spherical absorption correction.

Enter <CR> if none:

Average Friedel opposites in output file (Y or N) [Y]:

13g. IMPORTANT! Note well that the default will output only component 1. You can enter 0 for all data. Experience has shown, however, that only writing single or composite reflections to the .hkl file when a given component (e.g., 1) is present usually gives a better structure and reduces the statistical underestimate of the parameter esd's.

Output only reflections containing component N (0 to output all) [1]: 0

12329 Corrected reflections written to file twin_5.hkl

Reflections merged according to point-group 2/m

Ratio of minimum to maximum apparent transmission: 0.807218

14. Typically accept the default (Q) and exit twinabs.

It is now possible to generate a HKLF 4 format file with crude intensities for structure solution or a more accurate HKLF 5 format file for refinement. Repeat (R), write .hkl file (4 or 5), or quit (Q) [Q]:

Integration and scaling with the multicomponent .p4p file written by cell_now is complete. Use the standard APEX2 GUI and the twin_4.hkl data (typically copied or renamed to match the .ins file) to solve the structure.

Use the twin_5.hkl data (typically copied or renamed to match the .ins file) to refine the structure.

Appendix E. Config

The APEX2 Suite includes the ability to customize many features of the program. Most customization should be left to the site administrator, but some introduction is appropriate. The example below is a color-coded example of an actual Python file. It gives an introduction to configuring the program.

In the following text,

- Red denotes comments that result from # comments.
- Green denotes comments between triple quotes (""").
- Black denotes actual Python code.

Examples of configuration changes-

"""

The following is a collection of examples of possible changes to the configuration files for Apex2 and Proteum2. These files control the look and feel of the GUI.

Configuration variables the system administrator wants to set globally for all users go into a file called

bn-config.py that resides in C:\ for Windows or /usr/local/lib for Linux.

Individual users can set their own preferences

for most configuration variables in a file in their home directory.

This file is named bn-config.py for Windows

and .bn-config for Linux

(Under Windows, at a command prompt type

echo %UserProfile%

to find out where the user version of bn-config.py should be located.)

Additionally, configuration variables can be set for a specific

sample using a file in the sample directory

This file is bn-config for Windows and Linux

These files are read in the order given above so

values set globally are replaced by individual values and

global and individual values are replaced by specific values.

A few notes about Python comments.

Three quotation marks in a row on a line (""" start a comment section

The next three quotation marks (""") end the section

The text you have just read is in a comment section

"""

#end of commenting out a section with triple ""s

Pound or Number starts a simple comment. This can be anywhere on a line.

#

#

The default color scheme for the image display:

#

colorramp='Colored_SCD.ICM' # This is the typical BAXS orange display

#

""""

The most frequently used other color choices are:

Black_On_White_SCD.ICM

White_On_Black_SCD.ICM

The_Blues_ANY.ICM

Other color ramps are in:

Windows: C:\bn\src\gui\intensitycolormaps

Linux: /usr/local/bn/src/gui/intensitycolormaps

""""

#

```
# This is how user positions can be defined:
```

```
#
```

```
userpos1=
```

```
    {'name':'Zero@dx60','type': 'n',  
     'dx': 60.0,  
     'theta': 0,  
     'omega': 0,  
     'chi': 0,  
     'phi': +0.0 }
```

```
#
```

```
#If you want angles other than zero, the import projtls and del projtls must be used:
```

```
#
```

```
import projtls
```

```
userpos2=
```

```
    {'name':'Chi45@dx90','type': 'n',  
     'dx': 90.0,  
     'theta': -10.0*projtls.degrees,  
     'omega': -5.0*projtls.degrees,  
     'chi': 45.0*projtls.degrees,  
     'phi': 180.0*projtls.degrees }
```

```
del projtls
```

```
#
```

```
# Acceptable CCD temperature:
```

```
#
```

```
ccdtemperaturerange=(-65,-55)
```

```
#
```

```
# Auto connect to the instrument when needed:
#   (This is a protected variable that is controlled by the System Administrator)
#
autoconnect=1
#
# The following five values define matrix runs:
#
perpendicular_matrix_runs_2theta=30
# Set the 2-theta for the matrix measurement:
perpendicular_matrix_runs_distance=41
# Set the detector distance for the matrix measurement:
perpendicular_matrix_runs_nr_sweeps=3
# Do three sweeps or runs:
perpendicular_matrix_runs_sweep=6
# Each run will be 6 degrees long.
perpendicular_matrix_runs_frametime=None
# The frame time is left to be the default.
#
# Setup to get some debug messages for scans. This is useful if resume is failing.
#
#debug=('goniometer.scan',)
#debug=('expt.findresolution',)
#
#
```

```
# Hardware profile used by the data collection strategy planner:
bishost='bruker-server'
hardwareprofile='Kappa-APEX-II-euler.hrd'
#bishost='smart-apex'
#hardwareprofile='SMART-APEX-II.hrd'
#
#
# IP address or DNS name of the file server used for storing images,
# followed by the name of a network shared folder on that server.
# Add additional path segments as comma-separated and quoted strings.
# To find out the DNS name:
# For Linux type:   hostname      at the command prompt
# For Windows type: echo %computername%  at the DOS prompt.
#
fileserver=('x8-client','frames')

# Supported file name protocols. Currently these are 'smb' for
# files that are accessed over a MS Windows network and 'file' for
# local files.
universalfnprotocols=('smb','file')
#
```

Configuration of the APEX & PROTEUM programs

=====

The appearance and working of this suite of programs can be modified by a configuration file. The programs read the a seires of configuration files in folowing order. As described below, a specification in a later file overwrites a setting in an earlier file.

On unix these files are:

- 1 a file /usr/local/lib/bn-config.py
- 2 a file .bn-config in your home directory (Please note the initial "." character, which renders this file invisible for the normal "ls" command).
- 3 a file bn-config in your current directory.

On Windows these files are:

- 1 bn-config.py in the "SystemDrive" folder
- 2 bn-config.py in the "USERPROFILE" folder
- 3 bn-config in the current folder

If none of these files is present (the situation when the program has just been installed) all parameters are using built-in defaults. To change a single configuration parameter, creates or edits one of the three files and put in a value for the parameter to be customized.

You should put a parameter in (1) if it should be the system default for all users; put it in (2) if it is your own default, and other people might want to use other values; put it in (3) if it is only required for a single project.

The configuration files are read as "Python" programs, and therefore

syntax is very important. The best way to use it is to only use variable assignments or function calls, as described below. If required, you can comment out lines from your configuration file by preceding them with a '#' character.

Note:

- Leading space is significant. Start all assignments in the first column.
- Variable names are case sensitive.

Functionality groups

=====

Sometimes a complete group of options need to be changed to get a specific functionality. For these cases there are function calls that change a number of variables at once.

Due to the "default-like" nature of these function calls, they should probably be used only in the site-wide configuration files, and even there only at the beginning [but this is not enforced].

The available functions are:

default_protein()

This switches from the default "small molecule" parameters to more protein-like parameters. At this time, the following parameters are set:

```
chiralonly=1
autochiralpointgroup=1
resomode=1
resolution=2.5
```

default_noprotein()

This sets the defaults back to small molecule values

The following variables, in alphabetic order, can be set in a configuration file.

- * `anglemargin`, type=floating point, default=1.0
Do not change
- * `autochiralpointgroup`, type=boolean, default=0
This selects whether point groups are preferably chiral. This can be useful in a protein setting.
- * `autoconnect`, type=boolean, default=0
If this is set to 1, the connection to the BIS server will be created automatically without user-intervention whenever required. This variable can only be changed in the system-wide configuration file (see "protectedvariables")
- * `axcrit`, type=floating point angle, default=0.2 degrees
This is the criterion used by the automatic cell reduction algorithm to decide whether two axes coincide. Do not touch.
- * `badcolor`, type=tuple of three integers, default=(255,190,190)
Color in the GUI that is associated with 'bad' situations
- * `busycolor`, type=tuple of three integers, default=(240,240,96)
Color in the GUI that is associated with 'busy' situations
- * `bgdone`, type=tuple of three integers, default=(96,240,96)
Color in the GUI that is associated with 'done' situations
- * `bgerror`, type=tuple of three integers, default=(240,96,96)
Color in the GUI that is associated with 'error' situations

- * bgimpossible, type=tuple of three integers, default=(240,240,96)
Color in the GUI that is associated with 'impossible' situations
- * bgpossible, type=tuple of three integers, default=(96,240,96)
Color in the GUI that is associated with 'possible' situations
- * bgtodo, type=tuple of three integers, default=(96,240,96)
Color in the GUI that is associated with 'todo' situations
- * bgwarning, type=tuple of three integers, default=(240,240,96)
Color in the GUI that is associated with 'warning' situations
- * bishost, type=string, default is not set
The name or IP address of the BIS server computer
- * ccdtemperaturerange, type=tuple of 2 integers, default=(-60,-40)
Acceptable range (minimal,maximal) of temperatures in deg C for the detector
- * chiralonly, type=boolean, default=0
If set to 1, only chiral point groups will be used at any moment. Do not touch.
- * colorramp, type=file name, default='Colored_SCD.ICM'
Default color ramp used by the image display tool
- * communicationlog, type=file name, default='communication.log'
File name used for logging all communication between application and BIS. If unset, no logfile will be kept.

- * component1color, type=string (color name), default='green'
?
- * component2color, type=string (color name), default='blue'
?
- * component3color, type=string (color name), default='yellow'
?
- * datcoldx, type=floating point, default=35.0
Default sample-to-detector distance for data collections.
- * dbtype, type=string, default='postgresql'
Type of database engine used. Do not touch.
- * dbhost, type=string, default='127.0.0.1'
Name or IP address of the server running the database. The default is to run the database on the same host as the rest of the software, hence the default that refers to the local host.
- * dbport, type=string, default='5432'
IP port to use to address the database. Normally dependent on database installation.
- * dbname, type=string, default='testdb'
Name of the database. Do not touch.
- * debug, type=tuple of strings, default=()
This is a list of all modules that are to be debugged. This should not be touched except if a developer asks you to add something here to help track a problem.

- * defaulthardwaretype, type=integer, default=4
If different hardware types are supported, this can be used to select which one should be used. 4=BIS. Do not touch.
- * detectorsizemargin, type=floating point, default=0.1
Do not touch.
- * eventlogfile, type=file name, default='bn-event.log'
File in which important events are logged during program runs.
- * fontsize, type=integer, default=None
GUI font size in points
- * groupcolors, type=tuple of color names, default=('gray75',
 'red', 'green', 'yellow', 'blue', 'magenta', 'cyan', 'white',
 'gray25', 'brown', 'forest green',
 'khaki', 'midnight blue', 'violet', 'turquoise', 'gray50')
- * guicolor, type=tuple of three integers making a color,
default=0xd4,0xd0,0xc8
?
- * guistyle, type=string, default='windows'
The style of the GUI. Do not touch.
- * hardwareprofile, type=file name, default=None
The hardware profile to be used by COSMO for strategy calculations.
- * hashfilepattern, type=string, default='_##_####'
Do not touch

- * headerdisplay, type=?, default=not set
?
- * imageextension, type=string, default='.sfrm'
Do not touch
- * imagemask, type=int, default=0
value of a mask used in viewing powder frames
- * inhibitgc, type=boolean, default=0
Inhibit garbage collection. Do not touch.
- * localfilemap, type=map, default=None
A map of network drives, translating local file names to equivalent network names. On Windows machines, this is set automatically. On unix, this should represent a copy of the relevant parts of the samba configuration.
- * lowcurrent, type=floating point, default=5
Tube current in mA to set when an experiment finishes.
- * lowvoltage, type=floating point, default=20
Tube voltage in kV to set when an experiment finishes.
- * mailfrom, type=string, default=Not set
The "From" address of E-mails sent by the software (e.g. bug reports).
- * mailfromdomain, type=string, default=Not set
The domain of the From address of E-mails sent by the software. This will only be used if ''mailfrom'' is not set. The user name will be prepended.

- * `matrixstrategy`, type=list of scans, default=3 specific scans
The measurement strategy that is to be used to find a unit cell.
- * `minimalpowerfraction`, type=floating point, default=0.5
The minimal generator power as fraction of the maximal power that will be considered as sufficient to make a measurement
- * `mosaicity`, type=floating point angle, default=0.4
Expected normal width of a reflection, in degrees.
- * `nominalpowerfraction`, type=floating point, default=0.9
The normal generator power as fraction of the maximal power that the generator should be set to before a measurement
- * `nominalvoltage`, type=map, default={'MO':60,'CU':45,'AG':60}
A definition of the nominal tube voltage in kV as a function of the target element.
- * `orientdx`, type=floating point, default=-5
The default crystal to detector distance for the "aligned images" plugin. If given as a negative number, it is counted from the back of the dx-sled.
- * `orientdegreetime`, type=floating point, default=10.0
Default scan time in seconds per degree for an aligned image.
- * `orientscanwidth`, type=floating point, default=5.0
Default scan angle in degrees for an aligned image.
- * `overlapcolor`, type=color string, default='red'
?

- * overlaycolor, type=color string, default='green'
?
- * peakradiusfactor, type=floating point, default=2.2
How many times the single-sigma-spot radius should be used as
integration radius. Do not touch.
- * planes, type=string, default='0kl h0l hk0'
List of precession planes that is suggested as default planes for the
calculations
- * planethickness, type=floating point, default=0.1
Default thickness of precession images
- * precessionmargin, type=integer, default=1
How many pixels at the outer edge of the detector that is to be ignored
in precession calculations
- * prefereulerian, type=boolean, default=1
1 = prefer eulerian geometry over kappa geometry
0 = prefer kappa geometry over eulerian geometry
This should not be changed after installation.
- * preferredirection, type=integer, default=-1
?
- * productline, type=string, default='ANY'
A selection of the features in the program suite based on the type of
application we're running:
 'ANY' or 'ALL' = all products,
 'SCD' = single crystal,
 'XRD' = powder,
 'XRF' = fluorescence

- * `protectedvariables`, type=map,
default={'protectedvariables':1,'autoconnect':1}
A selection of variable names and a limit to which configuration files can be used to change them. This can be used to make sure users do not overwrite system defaults, like is done by default for the `autoconnect` variable.
- * `resolution`, type=floating point, default=0.77
Default resolution to go for in an experiment. 0.77 is reasonable for standard small-molecule experiments.
- * `scanrepr`, type=integer, default=-1
Determines how scans are represented in text:
 - 1: scan angle from x to y length y-x
 - 0: scan angle length y-x starting from x
 - 1: scan angle from x to y
- * `scanwidthtolerance`, type=floating point angle, default=0.003
Tolerance in degrees that determines whether two scans are equal. Do not touch.
- * `fileserver`, type=tuple of strings, default=()
Where experiment data files should be stored by default. First element in the tuple is the host name, second the share name, and the rest are successive folder names inside the share.
- * `fileserverprotocol`, type=string, default='smb'
The protocol that should be used to address the file server. Should be either 'file' (local file) or 'smb' (network drive)

- * `smbroot`, type=string, default='/smb'
Unix only. Where samba will mount shares made available from other hosts.
- * `smtphost`, type=string, default='localhost'
Host of a machine that can be addressed via SMTP to send E-mails.
- * `statuslines`, type=integer, default=1000
?
- * `temperaturerampspeed`, type=integer, default=240
The speed to use for ramping the temperature of a low-temperature device, in K per hour.
- * `universalfnprotocols`, type=tuple of strings, default=('smb','file')
Allowed protocols to exchange files with the BIS. Should be ('smb','file') when BIS is running on the same machine, and ('smb',) if BIS is running remotely.
- * `userpos1`, type=scan, default=Not set
?
- * `userpos2`, type=scan, default=Not set
?
- * `userpos3`, type=scan, default=Not set
?
- * `userpos4`, type=scan, default=Not set
?
- * `calibweaklevel`, type=integer, default=10
Minimal I/sigma for peaks to be used in calibration routines.

